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“The Right to Information, The Right to Live”

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Jawaharlal Nehru

“Step Out From the Old to the New”

IS 537 (2011): Toluene -Specification [PCD 3: Petroleum, Lubricants and their Related Products]

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“Knowledge is such a treasure which cannot be stolen”





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भारतीय मानक  
टॉलवीन — विशिष्टि  
( दूसरा पुनरीक्षण )

*Indian Standard*  
TOLUENE — SPECIFICATION  
( *Second Revision* )

ICS 71.080.15

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BUREAU OF INDIAN STANDARDS  
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## FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Petroleum, Lubricants and Their Related Products Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

This standard was first published on 1955 which was based on overseas national standards including the work accomplished till then by the Technical Committee, ISO/TC 78 — Aromatic Hydrocarbons, of International Organization for Standardization (ISO). It was revised in 1967 in order to cover toluene required with specially low content of non-sulphonable hydrocarbons. Besides, the requirement of distillation range was tightened in order to take care of the possibility of admixture with benzene. Colorimetric tests were also prescribed for acid wash test and colour. In this second revision, the requirements of residue on evaporation and corrosive sulphur have been deleted, and Gas Chromatographic (GC) method has been incorporated for determination of purity. Methods of test for distillation range and relative density have been modified.

Toluene is used as raw material in manufacture of alkylated, nitrated and halogenated organic intermediates for petrochemical dyestuffs and fine chemical industries. It is also used as solvent in paint, coating, adhesive and ink formulations.

Toluene is derived by suitable fractionation and refining by washing with acid or hydrorefining of crude benzole recovered from the gas produced during carbonization of coal in coke ovens and retorts or recovered as by-products in petroleum refining or petrochemical operations.

Earlier, toluene was essentially a coal base product which was being made available as by-product from coke ovens of steel plants. The requirements and methods of test were also stipulated on the basis of the publication by National Benzole and Allied Products Association (NBA) and the Standardization of Tar Products Tests Committee (STPC), U.K. in order to suit the prevailing quality of the product. However, the Committee took cognizance of the fact that consequent upon exploration of oil fields especially the Bombay High, substantial quantities of crude was being made available which has completely changed the scenario. There is a distinct shift in the production of various aromatic hydrocarbons from the coal base to petroleum base, as a result of which toluene is currently being made available in abundance which is more suitable for various purposes as compared to what was being made available from coal base. The present Committee, therefore, decided to update the standard in accordance with the latest development in the field and in the light of the experience gained. In this second revision, additional requirements of determination of purity by GC, non-aromatic hydrocarbons, benzene content, C-8 + aromatics and water content have been included. Requirement of residue on evaporation has been deleted.

IS 1839 : 1961 ‘Toluene, reagent grade’ has been withdrawn, as the Committee observed that this standard would cover the requirements of reagent grade toluene.

In this revision, considerable assistance has been drawn from the following standards published by International Organization for Standardization (ISO) and American Society for Testing and Materials (ASTM):

|                    |   |
|--------------------|---|
| ASTM D 841 : 1995  | Specification for nitration grade toluene   |
| ASTM D 2360 : 1995 | Trace impurities in monocyclic aromatic hydrocarbons by gas chromatography                          |
| ISO 4626 : 1980    | Volatile organic liquids — Determination of boiling range of organic solvents used as raw materials |
| ASTM D 848 : 1997  | Acid wash colour of industrial aromatic hydrocarbons  |
| ASTM D 847 : 1996  | Acidity of benzene, toluene, xylenes, solvent naphthas, and similar aromatic hydrocarbons           |
| ASTM D 849 : 1997  | Copper strip corrosion by industrial aromatic hydrocarbons  |
| ASTM D 853 : 1997  | Hydrogen sulphide and sulphur dioxide content (qualitative) of industrial aromatic hydrocarbons     |
| ASTM D 3505 : 1996 | Density or relative density of pure liquid chemicals  |

(Continued on third cover)

*Indian Standard*

# TOLUENE — SPECIFICATION

## ( Second Revision )

### **1 SCOPE**

This standard prescribes the requirements and the methods of sampling and test for toluene.

### **2 REFERENCES**

The following standards contain provisions which through reference in the text, constitute provisions of this standard. At the time of publication the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

| <i>IS No.</i>        | <i>Title</i>   |
|----------------------|--|
| 1070 : 1992          | Reagent grade water ( <i>third revision</i> )  |
| 1260 (Part 1) : 1973 | Pictorial marking for handling and labeling of goods: Part 1 Dangerous goods ( <i>first revision</i> ) |
| 1446 : 2002          | Classification of dangerous goods ( <i>second revision</i> )   |
| 5165 : 1969          | Interchangeable conical ground-glass joints  |
| 4644 : 1968          | Code of safety for benzene, toluene and xylene   |

| <i>IS No.</i> | <i>Title</i>  |
|---------------|---|
| 4905 : 1968   | Methods for random sampling   |
| 8768 : 2000   | Methods of measurement of colour in liquid chemical products platinum-cobalt scale ( <i>second revision</i> ) |

### **3 REQUIREMENTS**

The material shall comply with the requirements given in Table 1.

### **4 PACKING AND MARKING**

#### **4.1 Packing**

**4.1.1** The material shall be packed as agreed to between the purchaser and the supplier.

**4.1.2** All the containers in which the material is packed shall be dry, clean, and free from substances soluble in toluene and leak proof.

**4.1.3** The containers shall be securely closed, protected from light and shall be stored in a cool place.

**4.1.4** The containers for storage and transport of the material, since classified as flammable and dangerous goods, shall, in addition comply with the requirements

**Table 1 Requirements for Toluene**  
(*Clauses 3 and 7.1*)

| <b>Sl.<br/>No.<br/>(1)</b> | <b>Characteristic<br/>(2)</b>  | <b>Requirement<br/>(3)</b>   | <b>Methods of Test, Ref to Annex/<br/>IS/ASTM<br/>(4)</b> |
|----------------------------|--|--|---|
| i)                         | Toluene, percent by mass   | 99.8   | ASTM D 2360<br>(By gas chromatography)                    |
| ii)                        | Appearance   | Clear liquid free of sediment and haze when observed at 18.3 to 25.6°C | —   |
| iii)                       | Colour, platinum-cobalt scale, <i>Max</i>  | 10   | IS 8768   |
| iv)                        | Relative density at 15.56/15.56°C<br>or<br>Density, 20°C, g/ml   | 0.869-0.873  | A   |
| v)                         | Total sulphur, ppm, <i>Max</i>   | 0.865-0.870 <sup>1)</sup>  | B   |
| vi)                        | Distillation range including the temperature 110.6°C at 101.3 kPa (760 mm of Hg pressure), <i>Max</i> , °C | 0.6  | C   |
| vii)                       | Non-aromatic hydrocarbons, <i>Max</i> , percent by mass  | 0.1  | D   |
| viii)                      | Benzene content, mg/kg, <i>Max</i>   | 500 <sup>1)</sup>  | ASTM D 236  |
| ix)                        | Thiophene content  | 1 000  | ASTM D 4375   |
| x)                         | C 8 + Aromatics, ppm, <i>Max</i>   | Absent   | ASTM D 2360   |
| xi)                        | Hydrogen sulphide (H <sub>2</sub> S) and sulphurdioxide (SO <sub>2</sub> )                                 | 500  | E   |
| xii)                       | Water content, ppm   |  | By Karl Fischer method                                    |

NOTE — If purity by GC method [*see Sl No. (i)*] is carried out, distillation test is optional.

<sup>1)</sup> As agreed to between the purchaser and the supplier.

of the latest issue of Red Tariff and the requirements as laid down from time to time by the Chief Inspector of Explosives, Government of India, for packing, storage and transit of flammable liquids and the Board of Trade Regulations as applicable therein for transport by steamers.

**4.1.5** Necessary safeguards against the risk arising from the storage and handling of large volumes of flammable liquids (*see IS 1446*) shall be provided and all due precautions shall be taken [*see IS 1260 (Part 1)*] at all times to prevent accidents by fire or explosion.

**4.1.6** Except when they are opened for the purpose of cleaning and rendering them free from toluene vapour, all empty tanks or other containers shall be kept securely closed unless they have been cleaned and freed from toluene vapour.

## 4.2 Marking

**4.2.1** Each container shall be marked with the following information:

- a) Indication of the source of manufacture,
- b) Net mass of the material in the container,
- c) Batch number or code number, and
- d) Date of manufacture.

**4.2.2** Each container shall have the caution label 'FLAMMABLE' together with the corresponding symbol for labelling of dangerous goods as given in IS 1260 (Part1).

## 4.2.3 BIS Certification Marking

The containers may also be marked with the BIS Standard Mark.

**4.2.3.1** The use of the Standard Mark is governed by the provisions of *Bureau of Indian Standards Act, 1986* and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

## 5 HANDLING

Toluene is toxic and therefore it shall be handled carefully. Exposure of toluene in atmosphere should be monitored regularly. Persons exposed to toluene shall be periodically checked according to factory rules and local state regulations (*see IS 4644*).

## 6 SAMPLING

Representative samples of the material shall be prepared as prescribed in Annex F.

## 7 TEST METHODS

**7.1** Tests shall be conducted according to the methods prescribed in col 4 of Table 1.

## 7.2 Quality of Reagents

Unless otherwise specified, pure chemicals and distilled water (*see IS 1070*) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities, which affect the results of analysis.

## ANNEX A

[*Table 1, Sl No. (iv)*]

## DETERMINATION OF DENSITY OR RELATIVE DENSITY

**A-0** Two methods, pyknometer method and digital density meter method, have been specified. Pyknometer method shall be taken as reference method.

### A-1 OUTLINE OF THE PYKNOMETER METHOD

For materials listed in Table 2, the sample is drawn into a weighed and calibrated bicapillary pyknometer. The filler pyknometer is allowed to come to equilibrium at any convenient temperature between 10 and 30°C. The equilibrium temperature is measured to the nearest 0.02°C. The weight is determined by using a beam balance. The density, relative density,

or commercial density at the desired reference temperature is then calculated from the sample weight, a calibration factor proportional to an equal volume of water, and a multiplier which corrects for the buoyancy of air and the change in volume of the pyknometer and the sample due to deviation from the chosen reference temperature.

### A-2 APPARATUS

**A-2.1 Pyknometer**, 9 to 10 ml capacity, conforming to the dimensions given in Fig. 1, constructed of borosilicate glass, and having a total weight not exceeding 30 g.

**Table 2 20°C Reference Temperature Multiplier, F20, for Use in Computing Density  
(Clause A-1)**

| Temperature<br>°C<br>(1) | Benzene<br>(2) | Toluene<br>(3) | Mixed Xylenes<br>(4) | <i>o</i> -xylene<br>(5) | <i>m</i> -xylene<br>(6) | <i>p</i> -xylene<br>(7) |
|--------------------------|----------------|----------------|----------------------|-------------------------|-------------------------|-------------------------|
| 10.0                     | 0.988 22       | 0.989 41       | 0.990 28             | 0.990 52                | 0.990 28                | 0.990 11                |
| 10.2                     | 0.988 45       | 0.989 62       | 0.990 47             | 0.990 70                | 0.990 47                | 0.990 30                |
| 10.4                     | 0.988 65       | 0.989 83       | 0.990 66             | 0.990 89                | 0.990 66                | 0.990 49                |
| 10.6                     | 0.988 91       | 0.990 03       | 0.990 85             | 0.991 07                | 0.990 85                | 0.990 69                |
| 10.8                     | 0.989 14       | 0.990 24       | 0.991 04             | 0.991 26                | 0.991 04                | 0.990 66                |
| 11.0                     | 0.989 37       | 0.990 45       | 0.991 23             | 0.991 44                | 0.991 23                | 0.991 07                |
| 11.2                     | 0.989 60       | 0.990 66       | 0.991 42             | 0.991 63                | 0.991 42                | 0.991 26                |
| 11.4                     | 0.989 62       | 0.990 86       | 0.991 61             | 0.991 81                | 0.991 61                | 0.991 46                |
| 11.6                     | 0.990 05       | 0.991 07       | 0.991 79             | 0.992 00                | 0.991 79                | 0.991 65                |
| 11.8                     | 0.990 28       | 0.991 28       | 0.991 98             | 0.992 18                | 0.991 98                | 0.991 84                |
| 12.0                     | 0.990 51       | 0.991 48       | 0.992 17             | 0.992 37                | 0.992 17                | 0.992 04                |
| 12.2                     | 0.990 74       | 0.991 69       | 0.992 36             | 0.992 55                | 0.992 36                | 0.992 23                |
| 12.4                     | 0.990 97       | 0.991 90       | 0.992 55             | 0.992 74                | 0.992 53                | 0.992 42                |
| 12.6                     | 0.991 20       | 0.992 11       | 0.992 74             | 0.992 92                | 0.992 74                | 0.992 62                |
| 12.8                     | 0.991 44       | 0.992 31       | 0.992 93             | 0.993 11                | 0.992 93                | 0.992 81                |
| 13.0                     | 0.991 67       | 0.992 52       | 0.993 12             | 0.993 29                | 0.993 12                | 0.993 00                |
| 13.2                     | 0.991 90       | 0.992 73       | 0.993 31             | 0.993 48                | 0.993 31                | 0.993 20                |
| 13.4                     | 0.992 13       | 0.992 94       | 0.993 50             | 0.993 67                | 0.993 50                | 0.993 39                |
| 13.6                     | 0.992 36       | 0.993 15       | 0.993 69             | 0.993 85                | 0.993 69                | 0.993 58                |
| 13.8                     | 0.992 59       | 0.993 35       | 0.993 89             | 0.994 04                | 0.993 89                | 0.993 78                |
| 14.0                     | 0.992 82       | 0.993 56       | 0.994 08             | 0.994 22                | 0.994 08                | 0.993 97                |
| 14.2                     | 0.993 05       | 0.993 77       | 0.994 27             | 0.994 41                | 0.994 27                | 0.994 17                |
| 14.4                     | 0.993 29       | 0.993 98       | 0.994 46             | 0.994 60                | 0.994 46                | 0.994 36                |
| 14.6                     | 0.993 53       | 0.994 19       | 0.994 64             | 0.994 78                | 0.994 65                | 0.994 56                |
| 14.8                     | 0.993 75       | 0.994 40       | 0.994 84             | 0.994 97                | 0.994 84                | 0.994 75                |
| 15.0                     | 0.993 98       | 0.994 61       | 0.995 03             | 0.995 16                | 0.995 03                | 0.994 95                |
| 15.2                     | 0.994 21       | 0.994 81       | 0.995 22             | 0.995 34                | 0.995 22                | 0.995 14                |
| 15.4                     | 0.994 45       | 0.995 02       | 0.995 41             | 0.995 53                | 0.995 41                | 0.995 34                |
| 15.6                     | 0.994 68       | 0.995 23       | 0.995 61             | 0.995 72                | 0.995 61                | 0.995 53                |
| 15.8                     | 0.994 91       | 0.995 44       | 0.995 80             | 0.995 90                | 0.995 80                | 0.995 73                |
| 16.0                     | 0.995 15       | 0.995 65       | 0.995 99             | 0.996 09                | 0.995 99                | 0.995 92                |
| 16.2                     | 0.995 38       | 0.995 86       | 0.996 18             | 0.996 28                | 0.996 18                | 0.996 12                |
| 16.4                     | 0.995 61       | 0.996 07       | 0.996 37             | 0.996 46                | 0.996 37                | 0.996 31                |
| 16.6                     | 0.995 85       | 0.996 28       | 0.996 57             | 0.996 65                | 0.996 57                | 0.996 51                |
| 16.8                     | 0.996 08       | 0.996 49       | 0.996 76             | 0.996 84                | 0.996 76                | 0.996 70                |
| 17.0                     | 0.996 32       | 0.996 70       | 0.996 95             | 0.997 03                | 0.996 95                | 0.996 90                |
| 17.2                     | 0.996 55       | 0.996 91       | 0.997 14             | 0.997 21                | 0.997 14                | 0.997 10                |
| 17.4                     | 0.996 79       | 0.997 12       | 0.997 34             | 0.997 40                | 0.997 34                | 0.997 29                |
| 17.6                     | 0.997 02       | 0.997 33       | 0.997 53             | 0.997 59                | 0.997 53                | 0.997 49                |
| 17.8                     | 0.997 26       | 0.997 54       | 0.997 72             | 0.997 78                | 0.997 72                | 0.997 68                |
| 18.0                     | 0.997 49       | 0.997 75       | 0.997 91             | 0.997 97                | 0.997 91                | 0.997 88                |
| 18.2                     | 0.997 73       | 0.997 96       | 0.998 11             | 0.998 15                | 0.998 11                | 0.998 08                |
| 18.4                     | 0.997 96       | 0.998 17       | 0.998 30             | 0.998 34                | 0.998 30                | 0.998 27                |
| 18.6                     | 0.998 20       | 0.998 38       | 0.998 49             | 0.998 53                | 0.998 49                | 0.998 47                |
| 18.8                     | 0.998 43       | 0.998 59       | 0.998 69             | 0.998 72                | 0.998 69                | 0.998 67                |
| 19.0                     | 0.998 67       | 0.998 80       | 0.998 88             | 0.998 91                | 0.998 88                | 0.998 86                |
| 19.2                     | 0.998 90       | 0.999 01       | 0.999 07             | 0.999 10                | 0.999 07                | 0.999 06                |
| 19.6                     | 0.999 38       | 0.999 43       | 0.999 46             | 0.999 47                | 0.999 46                | 0.999 46                |
| 19.8                     | 0.999 61       | 0.999 64       | 0.999 66             | 0.999 66                | 0.999 66                | 0.999 65                |
| 20.0                     | 0.999 85       | 0.999 85       | 0.999 85             | 0.999 85                | 0.999 85                | 0.999 85                |
| 20.2                     | 1.000 09       | 1.000 06       | 1.000 04             | 1.000 04                | 1.000 04                | 1.000 05                |
| 20.4                     | 1.000 32       | 1.000 27       | 1.000 24             | 1.000 23                | 1.000 24                | 1.000 25                |
| 20.6                     | 1.000 56       | 1.000 48       | 1.000 43             | 1.000 42                | 1.000 43                | 1.000 44                |
| 20.8                     | 1.000 80       | 1.000 69       | 1.000 63             | 1.000 61                | 1.000 63                | 1.000 64                |
| 21.0                     | 1.001 04       | 1.000 91       | 1.000 82             | 1.000 80                | 1.000 82                | 1.000 84                |
| 21.2                     | 1.001 28       | 1.001 12       | 1.001 02             | 1.000 99                | 1.001 02                | 1.001 04                |
| 21.4                     | 1.001 51       | 1.001 33       | 1.001 21             | 1.001 18                | 1.001 21                | 1.001 24                |
| 21.6                     | 1.001 75       | 1.001 54       | 1.001 41             | 1.001 37                | 1.001 41                | 1.001 43                |
| 21.8                     | 1.001 99       | 1.001 75       | 1.001 60             | 1.001 56                | 1.001 60                | 1.001 63                |
| 22.0                     | 1.002 23       | 1.001 96       | 1.001 80             | 1.001 75                | 1.001 80                | 1.001 63                |
| 22.2                     | 1.002 47       | 1.002 18       | 1.001 99             | 1.001 94                | 1.001 99                | 1.002 03                |
| 22.4                     | 1.002 71       | 1.002 39       | 1.002 19             | 1.002 13                | 1.002 19                | 1.002 23                |
| 22.6                     | 1.002 95       | 1.002 60       | 1.002 38             | 1.002 32                | 1.002 38                | 1.002 43                |
| 22.8                     | 1.003 19       | 1.002 81       | 1.002 58             | 1.002 51                | 1.002 58                | 1.002 63                |
| 23.0                     | 1.003 42       | 1.003 02       | 1.002 78             | 1.002 70                | 1.002 78                | 1.002 83                |
| 23.2                     | 1.003 66       | 1.003 24       | 1.002 97             | 1.002 89                | 1.002 97                | 1.003 03                |

**Table 2 (Concluded)**

| Temperature<br>°C<br>(1) | Benzene<br>(2) | Toluene<br>(3) | Mixed Xylenes<br>(4) | <i>o</i> -xylene<br>(5) | <i>m</i> -xylene<br>(6) | <i>p</i> -xylene<br>(7) |
|--------------------------|----------------|----------------|----------------------|-------------------------|-------------------------|-------------------------|
| 23.4                     | 1.003 90       | 1.003 45       | 1.003 17             | 1.003 08                | 1.003 17                | 1.003 22                |
| 23.6                     | 1.004 14       | 1.003 66       | 1.003 36             | 1.003 27                | 1.003 36                | 1.003 42                |
| 23.8                     | 1.004 38       | 1.003 87       | 1.003 56             | 1.003 46                | 1.003 56                | 1.003 62                |
| 24.0                     | 1.004 62       | 1.004 09       | 1.003 76             | 1.003 65                | 1.003 76                | 1.003 82                |
| 24.2                     | 1.004 87       | 1.004 30       | 1.003 95             | 1.003 84                | 1.003 95                | 1.004 02                |
| 24.4                     | 1.005 11       | 1.004 51       | 1.004 15             | 1.004 03                | 1.004 15                | 1.004 22                |
| 24.6                     | 1.005 35       | 1.004 73       | 1.004 35             | 1.004 22                | 1.004 35                | 1.004 42                |
| 24.8                     | 1.005 59       | 1.004 94       | 1.004 54             | 1.004 42                | 1.004 54                | 1.004 62                |
| 25.0                     | 1.005 83       | 1.005 15       | 1.004 74             | 1.004 61                | 1.004 74                | 1.004 82                |
| 25.2                     | 1.006 07       | 1.005 37       | 1.004 94             | 1.004 80                | 1.004 94                | 1.005 02                |
| 25.4                     | 1.006 31       | 1.005 58       | 1.005 14             | 1.004 99                | 1.005 14                | 1.005 22                |
| 25.6                     | 1.006 56       | 1.005 79       | 1.005 33             | 1.005 18                | 1.005 33                | 1.005 42                |
| 25.8                     | 1.006 80       | 1.006 01       | 1.005 53             | 1.005 37                | 1.005 53                | 1.005 63                |
| 26.0                     | 1.007 04       | 1.006 22       | 1.005 73             | 1.005 57                | 1.005 73                | 1.005 83                |
| 26.2                     | 1.007 28       | 1.006 43       | 1.005 93             | 1.005 76                | 1.005 93                | 1.006 03                |
| 26.4                     | 1.007 53       | 1.006 65       | 1.006 12             | 1.005 95                | 1.006 12                | 1.006 23                |
| 26.6                     | 1.007 77       | 1.006 86       | 1.006 32             | 1.006 14                | 1.006 32                | 1.006 43                |
| 26.8                     | 1.008 01       | 1.007 07       | 1.006 52             | 1.006 34                | 1.006 52                | 1.006 63                |
| 27.0                     | 1.008 25       | 1.007 29       | 1.006 72             | 1.006 53                | 1.006 72                | 1.006 83                |
| 27.2                     | 1.008 50       | 1.007 50       | 1.006 92             | 1.006 72                | 1.006 92                | 1.007 03                |
| 27.4                     | 1.008 74       | 1.007 72       | 1.007 11             | 1.006 91                | 1.007 11                | 1.007 24                |
| 27.6                     | 1.008 99       | 1.007 93       | 1.007 31             | 1.007 11                | 1.007 31                | 1.007 44                |
| 27.8                     | 1.009 23       | 1.008 15       | 1.007 51             | 1.007 30                | 1.007 51                | 1.007 64                |
| 28.0                     | 1.009 47       | 1.008 36       | 1.007 71             | 1.007 49                | 1.007 71                | 1.007 84                |
| 28.2                     | 1.009 72       | 1.008 58       | 1.007 91             | 1.007 69                | 1.007 91                | 1.008 04                |
| 28.4                     | 1.009 96       | 1.008 79       | 1.008 11             | 1.007 88                | 1.008 11                | 1.008 25                |
| 28.6                     | 1.010 21       | 1.009 01       | 1.008 31             | 1.008 07                | 1.008 31                | 1.008 45                |
| 28.8                     | 1.010 45       | 1.009 22       | 1.008 51             | 1.008 27                | 1.008 51                | 1.008 65                |
| 29.0                     | 1.010 70       | 1.009 44       | 1.008 71             | 1.008 46                | 1.008 71                | 1.008 85                |
| 29.2                     | 1.010 94       | 1.009 65       | 1.008 91             | 1.008 66                | 1.008 91                | 1.009 06                |
| 29.4                     | 1.011 19       | 1.009 87       | 1.009 11             | 1.008 85                | 1.009 11                | 1.009 26                |
| 29.6                     | 1.011 43       | 1.010 08       | 1.009 31             | 1.009 04                | 1.009 31                | 1.009 46                |
| 29.8                     | 1.011 68       | 1.010 30       | 1.009 51             | 1.009 24                | 1.009 51                | 1.009 66                |
| 30.0                     | 1.011 92       | 1.010 51       | 1.009 71             | 1.009 43                | 1.009 71                | 1.009 87                |

NOTE — Choose a multiplier for the material being measured corresponding to the bath Temperature at which the Pyknometer is equilibrated.

**A-2.2 Bath**, having a depth of at least 300 mm, capable of being maintained constant to  $\pm 0.02^\circ\text{C}$  at any convenient temperature between  $10^\circ\text{C}$  and  $30^\circ\text{C}$ . Provide a support for the pyknometer (*see Fig. 2*) constructed of any suitable non-corrosive metal.

**A-2.3 Bath Thermometer**, having a range from  $-8$  to  $+32^\circ\text{C}$ .

### A-3 PREPARATION OF APPARATUS

#### A-3.1 Acid Cleaning

When liquid fails to drain cleanly from the walls of the pyknometer or its capillary. Clean with hot chromic acid solution, thoroughly and rinse well with water. Dry at  $105^\circ\text{C}$  to  $110^\circ\text{C}$  for at least 1 h, preferably with a slow current of filtered air passing through the pyknometer.

#### A-3.2 Solvent Cleaning

For use between determinations. Rinse with toluene and then with anhydrous acetone, drying with a filtered stream of dry air.

### A-4 CALIBRATION OF APPARATUS

**A-4.1** Using the procedure described in **A-5**, determine the weight of freshly boiled reagent water held by the pyknometer with the water level at each of three different scale points on the graduated arms. Make all weighings on the same day, using the same balance and weights.

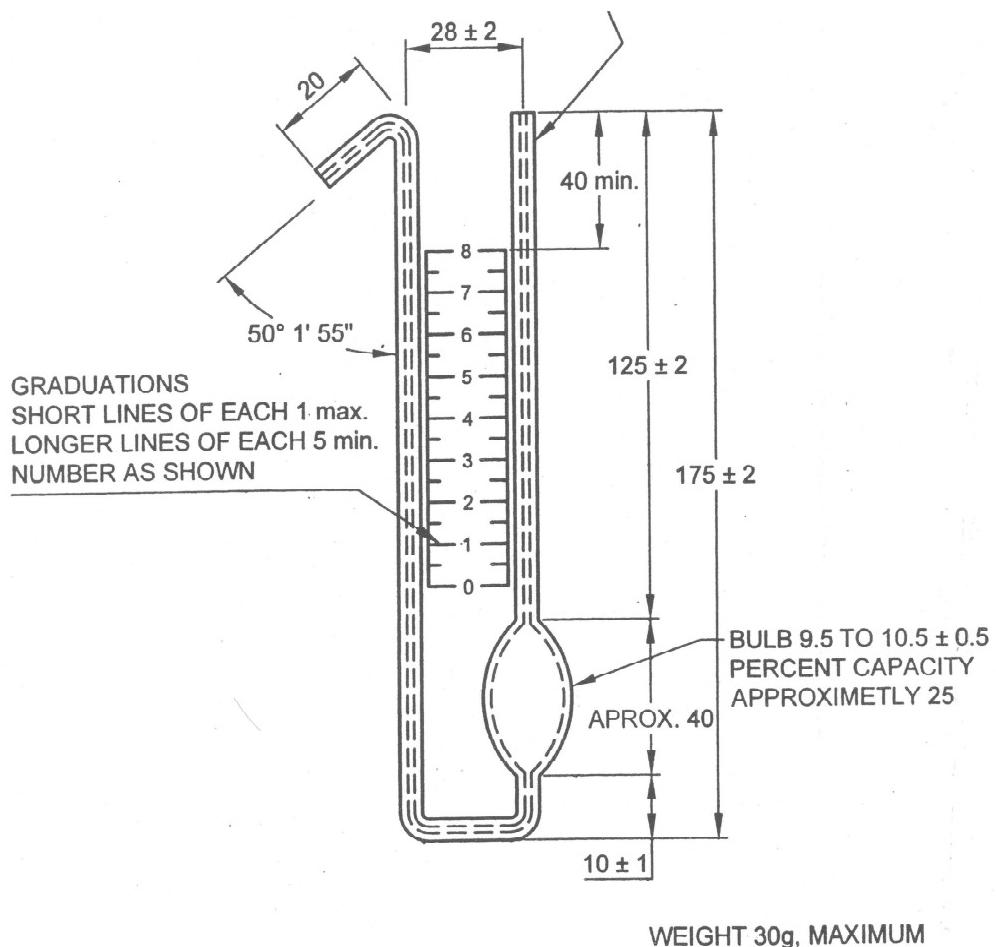
**A-4.2** Calculate the volume,  $V_T^P$ , at each scale point tested by means of the following equation; carry all calculations in 6 non-zero digits and round to 4 decimal places:

Pyknometer capacity,  $V_T^P$ , ml =  $A(W^w/d_t^w) + B(T - t)$   
where

$A$  = air buoyancy coefficient, a constant for the temperature range involved = 1.001 064;

$V_T^P$  = volume of pyknometer at reference temperature,  $T$ ;

$W^w$  = weight of water in air, contained in the pyknometer, in g;



NOTE — The graduation lines shall extend around the entire circumference of the pyknometer at the integral numbers 0, 1, 2 cm etc... half way around at the half divisions 0.5, 1.5, etc... and shorter lines for the intermediate subdivisions.

All dimensions in millimetres.

FIG. 1 PYKNOMETER

$d_t^w$  = density of water at  $t$  (see Table 6);

$t$  = temperature, °C;

$T$  = reference temperature, 20°C or 15.56°C; and

$B$  = volumetric coefficient of expansion of 9.5 ml of a borosilicate glass pyknometer,  $9.262\ 76 \times 10^{-5}$  ml/°C.

**A-4.3** Prepare a calibration curve by plotting apparent volume,  $V_A$  (the sum of the scale readings on the two arms of the pyknometer) against the corresponding calculated volume,  $V_T^P$ . If a straight line cannot be drawn through the three points, discard the data and determine three additional points so that a straight calibration line can be drawn, such that no data point lies more than 0.000 2 ml units from the line. If neither set of data meets the condition, the diameters of the graduated capillary arms are not sufficiently uniform, and the pyknometer shall be discarded.

**A-4.4** From the curve obtained, prepare a table of

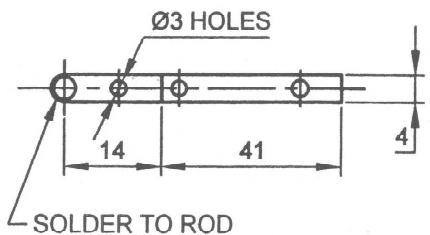
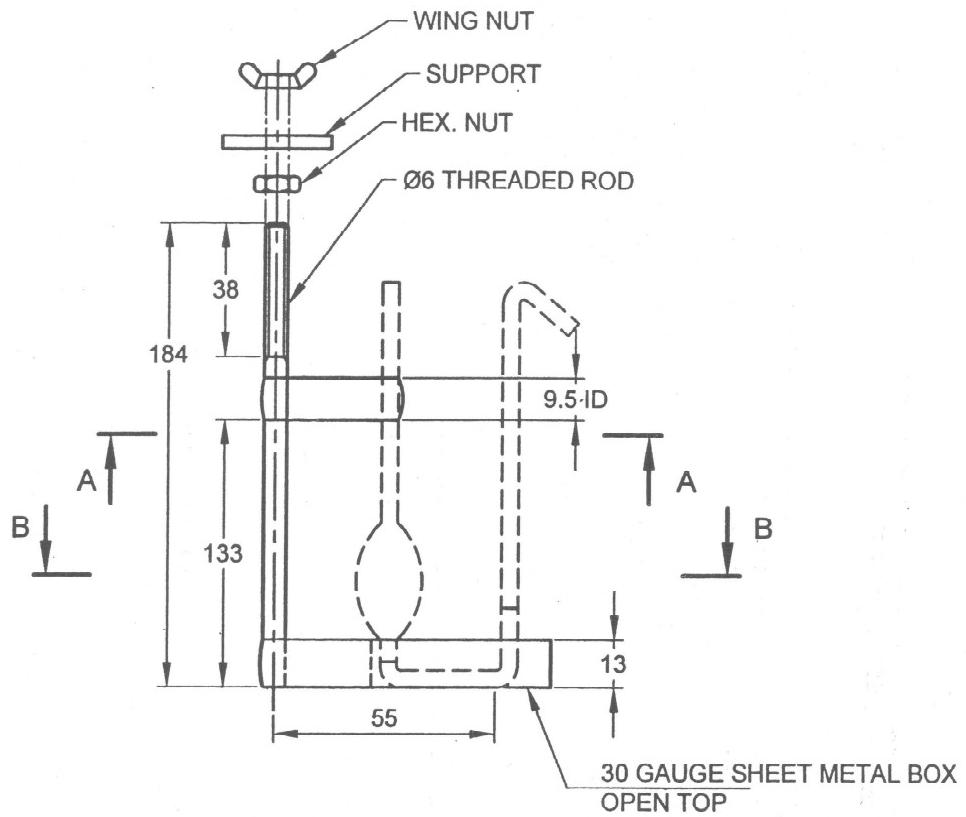
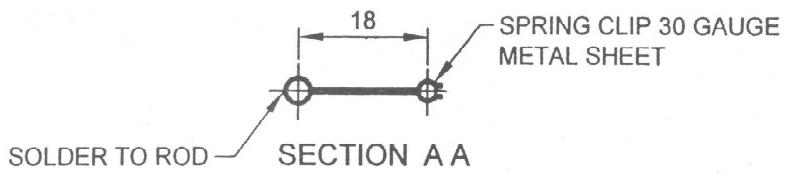
apparent volume,  $V_A$  (sum of scale readings of both arms), as apparent volume against corresponding calculated volumes,  $V_T^P$ , in increments of 0.000 1 ml. Label Table 3 with the reference temperature to which it applies.

#### A-5 PROCEDURE

**A-5.1** Weigh the clean, dry pyknometer to 0.1 mg and record the weight.

**A-5.2** With the sample at approximately the test temperature, fill the pyknometer by holding it in an upright position and placing the hooked tip in the sample; the liquid will then be drawn over the bend in the capillary by surface tension. Allow the pyknometer to fill by siphoning (about 1 min) and break the siphon when the liquid level in the bulb arm of the pyknometer reaches the lowest graduation mark.

**A-5.3** Thoroughly dry the wet tip. Wipe the body of



SECTION B-B

All dimensions in millimetres.

FIG. 2 PYKNOMETER HOLDER

the pyknometer with a chemically clean, lint-free cloth slightly damp with water (*see Note*) and weigh the filled pyknometer to the nearest 0.1mg.

**NOTE**—In atmospheres below 60 percent relative humidity, drying the pyknometer by rubbing with a dry cotton cloth will induce static charges equivalent to a loss of about 1 mg or more in the weight of the pyknometer. This charge may not be completely dissipated in less than 30 min, and can be detected by touching the pyknometer to the wire hook in the balance and then drawing it away slowly. If the pyknometer exhibits an attraction for the wire hook, it may be considered to have a static charge.

**A-5.4** Place the pyknometer in the holder in a constant temperature bath held at any convenient temperature 10°C and 30°C within  $\pm 0.02^\circ\text{C}$ . When the liquid level has reached temperature equilibrium (usually in about 10 min) and while still in the water bath, read the scale to the nearest 0.2 small divisions at the liquid level in each arm.

#### A-6 CALCULATION

Compute the density or relative density, or both, by means of the following equations:

$$\text{Density, g/ml at } 20^\circ\text{C} = (W^s / V_{20}^p) \times F_{20} + 0.001\ 21$$

$$\text{Relative density at } 20/20^\circ\text{C} = (W^s / V_{20}^p) \times F_{20} + (0.001\ 21) / 1.000\ 96$$

where

$W^s$  = observed weight of sample, corrected for variation of weights, g,

$V_{20}^p$  = calculated volume,  $V_T^p$ , of sample at  $20^\circ\text{C}$ , millilitres, obtained from the pyknometer calibration table, and

$F_{20}$  = constants taken from Table 2, corresponding to the test temperature.

#### A-7 OUTLINE OF DIGITAL DENSITY METER METHOD

A small volume (approximately 0.7 ml) of liquid sample is introduced into an oscillating sample tube and the change in oscillating frequency caused by the change in the mass of the tube is used in conjunction with calibration data to determine the density of the sample.

#### A-8 APPARATUS

**A-8.1 Digital Density Analyzer** — A digital analyzer consisting of a U-shaped, oscillating sample tube and a system for electronic excitation, frequency counting, and display. The analyzer must accommodate the accurate measurement of the sample temperature during measurement or must control the sample temperature as described in **A-8.2**. The instrument shall be capable of meeting the precision requirements described in this test method.

**A-8.2 Circulating Constant-Temperature Bath (Optional)**, capable of measuring the temperature of the circulating liquid constant to  $\pm 0.05^\circ\text{C}$  in the desired range. Temperature control can be maintained as part of the density analyzer instrument package.

**A-8.3 Syringes** — At least 2 ml in volume with a tip or an adapter tip that will fit the opening of the oscillating tube.

**A-8.4 Flow-Through of Pressure Adapter** for use as an alternative means of introducing the sample into the density analyzer either by a pump or by vacuum.

**A-8.5 Thermometer**, calibrated and graduated to  $0.1^\circ\text{C}$ , and a thermometer holder that can be attached to the instrument for setting and observing the test temperature. In calibrating the thermometer, the ice

**Table 3 Density of Water, g/ml**  
(Clause A-4.4)

| $t, ^\circ\text{C}$ | 0.0   | 0.1 | 0.2 | 0.3 | 0.4 | 0.5 | 0.56 | 0.6 | 0.7 | 0.8 | 0.9 |
|---------------------|-------|-----|-----|-----|-----|-----|------|-----|-----|-----|-----|
| 15                  | 0.999 | 13  | 11  | 10  | 08  | 07  | 05   | 04  | 02  | 00  | 99  |
| 16                  | 0.998 | 97  | 96  | 94  | 92  | 91  | 89   | 87  | 86  | 84  | 82  |
| 17                  |       | 80  | 79  | 77  | 75  | 73  | 72   | 70  | 68  | 66  | 64  |
| 18                  |       | 62  | 61  | 89  | 57  | 55  | 53   | 51  | 49  | 47  | 45  |
| 19                  |       | 43  | 42  | 40  | 38  | 36  | 34   | 32  | 30  | 27  | 25  |
| 20                  |       | 23  | 21  | 19  | 17  | 15  | 13   | 11  | 09  | 07  | 04  |
| 21                  |       | 02  | 00  | 98  | 96  | 93  | 91   | 89  | 87  | 85  | 82  |
| 22                  | 0.997 | 80  | 78  | 75  | 73  | 71  | 69   | 66  | 64  | 62  | 59  |
|                     |       | 57  | 64  | 52  | 50  | 47  | 45   | 42  | 40  | 38  | 35  |
| 23                  |       | 33  | 30  | 28  | 25  | 23  | 20   | 18  | 15  | 13  | 10  |
| 24                  |       | 08  | 05  | 02  | 00  | 97  | 95   | 92  | 89  | 87  | 84  |
| 25                  |       | 81  | 79  | 76  | 73  | 71  | 68   | 65  | 63  | 60  | 57  |
| 26                  |       | 54  | 52  | 49  | 46  | 43  | 41   | 38  | 35  | 32  | 29  |
| 27                  |       | 26  | 24  | 21  | 18  | 15  | 12   | 09  | 06  | 03  | 00  |
| 28                  |       | 98  | 95  | 92  | 89  | 86  | 83   | 80  | 77  | 74  | 72  |
| 29                  | 0.995 | 68  | 65  | 62  | 59  | 56  | 53   | 50  | 46  | 43  | 40  |
| 30                  |       |     |     |     |     |     |      |     |     |     |     |

point and bore connections should be estimated to the nearest 0.05°C.

#### A-9 REAGENTS AND MATERIALS

**A-9.1 Water**, redistilled freshly boiled and cooled reagent water for use as a primary calibration standard.

**A-9.2 Petroleum Naphtha**, for flushing viscous petroleum samples from the sample tube (extremely flammable).

**A-9.3 Acetone**, for flushing and drying the sample tube (extremely flammable).

**A-9.4 Dry Air**, for blowing the oscillator tube.

#### A-10 PREPARATION OF APPARATUS

Set up the density analyzer and constant temperature bath following the manufacturer's instructions. Adjust the bath or internal temperature control so that the desired test temperature is established and maintained in the sample compartment of the analyzer. Calibrate the instrument at the same temperature at which the density of the sample is to be measured.

NOTE — Precise setting and control of the test temperature in the sample tube is extremely important. An error of 0.1 °C can result in a change in density of one in the fourth decimal.

#### A-11 CALIBRATION OF APPARATUS

**A-11.1** Calibrate the instrument when first set up and whenever the test temperature is changed. Thereafter, conduct calibration checks at weekly intervals during routine operation.

**A-11.2** Initial calibration, or calibration after a change in test temperature, necessitates calculation of the values of the constants *A* and *B* from the periods of oscillation (*T*) observed when the sample cell contains air and redistilled, freshly boiled and cooled reagent water. Other calibrating materials such as *n*-nonane, *n*-tridecane, cyclohexane, and *n*-hexadecane (for high temperature applications) can also be used as appropriate.

**A-11.2.1** While monitoring the oscillator period, *T* flush the sample tube with petroleum naphtha, followed with an acetone flush and dry with dry air. Contaminated or humid air can affect the calibration. When these conditions exist in the laboratory, pass the air used for calibration through a suitable purification and drying train. In addition, the inlet and outlet ports for the U-tube must be plugged during measurement of the calibration air to prevent ingress of moist air.

**A-11.2.2** Allow the dry air in the U-tube to come to thermal equilibrium with the test temperature and record the *T*-value for air.

**A-11.2.3** Introduce a small volume (about 0.7 ml) of redistilled, freshly boiled and cooled reagent water into the sample tube from the bottom opening using a suitable syringe. The test portion must be homogeneous and free of even the smallest air or gas bubbles. The sample tube does not have to be completely full as long as the liquid meniscus is beyond the suspension point. Allow the display to reach a steady reading and record the *T*-value for water.

**A-11.2.4** Calculate the density of air at the temperature of test using the following equation:

$$D_a, \text{ g/ml} = 0.001\ 293[273.15/T][P/760]$$

where

*T* = temperature, in K, and

*P* = barometric pressure, in torr.

**A-11.2.5** Determine the density of water at the temperature of test by reference to Table 4.

**A-11.2.6** Using the observed *T* values and the reference values for water and air, calculate the values of the constants *A* and *B* using the following equations:

$$A = [T_w^2 - T_a^2]/[d_w - d_a]$$

$$B = T_a^2 \times (A \times d_a)$$

where

*T<sub>w</sub>* = observed period of oscillation for cell containing water,

*T<sub>a</sub>* = observed period of oscillation for cell containing air,

*d<sub>w</sub>* = density of water at test temperature, and

*d<sub>a</sub>* = density of air at test temperature.

Alternatively, use the *T* and *d* values for the other reference liquid, if one is used.

**A-11.2.7** If the instrument is equipped to calculate density from the constants *A* and *B* and the observed *T*-value from the sample, then enter the constants in the instrument memory in accordance with the manufacturer's instructions.

**A-11.2.8** Check the calibration and adjust if needed by performing the routine calibration check described in A-11.3.

**A-11.2.9** To calibrate the instrument to display relative density, that is, the density of the sample at a given temperature referred to the density of water at the same temperature, follow sections A-11.2.1 through A-11.2.7, but substitute 1.000 for *d<sub>w</sub>* in performing the calculations described in A-11.2.6.

**A-11.3** Weekly calibration adjustments to constants *A* and *B* can be made if required, without repeating the calculation procedure.

**NOTE** — The need for a change in calibration is generally attributable to deposits in the sample tube that are not removed by the routine flushing procedure. Although this condition can be compensated for by adjusting *A* and *B*, it is good practice to clean the tube with warm chromic acid solution\* whenever a major adjustment is required. Chromic acid solution is the most effective cleaning agent; however, surfactant cleaning fluids have also been used successfully.

\*Warning — Causes severe burns. A recognized carcinogen.

**A-11.3.1** Flush and dry the sample tube as described in **A-11.2.1** and allow the display to reach a steady reading. If the display does not exhibit the correct density for air at the temperature of test, repeat the cleaning procedure or adjust the value of constant *B* commencing with the last decimal place until the correct density is displayed.

**A-11.3.2** If adjustment to constant *B* was necessary in **A-11.3.1**, then continue the recalibration by introducing redistilled, freshly boiled and cooled reagent water into the sample tube as described in **A-11.2.3** and allow the display to reach a steady reading. If the instrument has been calibrated to display the density, adjust the reading to the correct value for water at the test temperature (see Table 4) by changing the value of constant *A*, commencing with the last decimal place. If the instrument has been calibrated to display the relative density, adjust the reading to the value 1.000 0.

**NOTE** — In applying this weekly calibration procedure, it can be found that more than one value each for *A* and *B*, differing in the fourth decimal place, will yield the correct density reading for the density of air and water. The setting chosen would then be dependent upon whether it was approached from a higher or lower value. The setting selected by this method could have the effect of altering the fourth place of the reading obtained for a sample.

**A-11.4** Some analyzer models are designed to display the measured period of oscillation only (*T*-values) and their calibration requires the determination of an instrument constant *K*, which must be used to calculate the density or relative density from the observed data.

**A-11.4.1** Flush and dry the sample tube as described in **A-11.2.1** and allow the display to reach a steady reading. Record the *T*-value for air.

**A-11.4.2** Introduce redistilled, freshly boiled and cooled reagent water into the sample tube as described in **A-11.2.3**, allow the display to reach a steady reading and record the *T*-value for water.

**A-11.4.3** Using the observed *T*-values and the reference values for water and air (see **A-11.2.4** and **A-11.2.5**), calculate the instrument constant *K* using the following equations:

For density:

$$K_1 = [d_w - d_a]/[T_w^2 - T_a^2]$$

For relative density:

$$K_2 = [1.000 0 - d_a]/[T_w^2 - T_a^2]$$

where

*T<sub>w</sub>* = observed period of oscillation for cell containing water,

*T<sub>a</sub>* = observed period of oscillation for cell containing air,

*d<sub>w</sub>* = density of water at test temperature, and

*d<sub>a</sub>* = density of air at test temperature.

## A-12 PROCEDURE

**A-12.1** Introduce a small amount (about 0.7 ml) of sample into the clean, dry sample tube of the instrument using a suitable syringe.

**A-12.2** The sample can also be introduced by siphoning. Plug the external TFE-fluorocarbon capillary tube into the lower entry port of the sample tube. Immerse the other end of the capillary in the sample and apply suction to the upper entry port using a syringe or vacuum line until the sample tube is properly filled.

**A-12.3** Turn on the illumination light and examine

**Table 4 Density of Water**  
(Clause A-11.2.5 and A-11.3.2)

| Temperature, °C<br>(1) | Density, g/ml<br>(2) | Temperature, °C<br>(1) | Density, g/ml<br>(2) | Temperature, °C<br>(1) | Density, g/ml<br>(2) |
|------------------------|----------------------|------------------------|----------------------|------------------------|----------------------|
| 0.0                    | 0.999 840            | 21.0                   | 0.997 991            | 40.0                   | 0.992 212            |
| 3.0                    | 0.999 964            | 22.0                   | 0.997 769            | 45.0                   | 0.990 208            |
| 4.0                    | 0.999 972            | 23.0                   | 0.997 537            | 50.0                   | 0.988 030            |
| 5.0                    | 0.999 964            | 24.0                   | 0.997 295            | 55.0                   | 0.985 688            |
| 10.0                   | 0.999 699            | 25.0                   | 0.997 043            | 60.0                   | 0.983 191            |
| 15.0                   | 0.999 099            | 26.0                   | 0.996 782            | 65.0                   | 0.980 546            |
| 15.56                  | 0.999 012            | 27.0                   | 0.996 511            | 70.0                   | 0.977 759            |
| 16.0                   | 0.998 943            | 28.0                   | 0.996 231            | 75.0                   | 0.974 837            |
| 17.0                   | 0.998 774            | 29.0                   | 0.995 943            | 80.0                   | 0.971 785            |
| 18.0                   | 0.998 595            | 30.0                   | 0.995 645            | 85.0                   | 0.968 606            |
| 19.0                   | 0.998 404            | 35.0                   | 0.994 029            | 90.0                   | 0.965 305            |
| 20.0                   | 0.998 203            | 37.78                  | 0.993 042            | 100                    | 0.958 345            |

the sample tube carefully. Make sure that no bubbles are trapped in the tube, and that it is filled to just beyond the suspension point on the right-hand side. The sample must be homogeneous and free of even the smallest bubbles.

**NOTE** — If the sample is too dark in colour to determine the absence of bubbles with certainty, the density cannot be measured within the stated precision limits.

**A-12.4** Turn the illumination light off immediately after sample introduction, because the heat generated can affect the measurement temperature.

**A-12.5** After the instrument displays a steady reading to four significant figures for density and five for  $T$ -values, indicating that temperature equilibrium has been reached, record the density or  $T$ -value.

## A-13 CALCULATION

### A-13.1 Calculating Density Analyzers

The recorded value is the final result, expressed either as density, in g/ml or kg/m<sup>3</sup>, or as relative density.

**NOTE** — 1 000 kg/m<sup>3</sup> = 1 g/ml.

**A-13.2 Non-calculating Density Analyzers** using the observed  $T$ -value for the sample and the  $T$ -value for water and appropriate instrument constants determined in **A-11.4.3**, calculate the density or relative density using the equation given below. Carry out all calculations to six significant figures and round the final results to four.

For density:

$$\text{Density, g/ml (kg/dm}^3\text{) at } t = d_w + K_1 (T_s^2 - T_w^2)$$

For relative density:

$$\text{Relative density, } t/t = 1 + K_2 (T_s^2 - T_w^2)$$

where

$T_w$  = observed period of oscillation for cell containing water,

$T_s$  = observed period of oscillation for cell containing sample,

$d_w$  = density of water at test temperature,

$K_1$  = instrument constant for density,

$K_2$  = instrument constant for relative density, and

$t$  = temperature of test, °C.

## ANNEX B

[Table 1, Sl No. (v)]

### DETERMINATION OF TOTAL SULPHUR CONTENT

#### B-1 GENERAL

##### B-1.1 Outline of the Method

The sample is reacted with Raney nickel. The hydrogen sulphide liberated from the nickel sulphide thus formed is absorbed and titrated with mercuric acetate. Certain oxygenated sulphur compounds are not completely determined and some olefins interfere with the test.

**NOTE** — Stringent precautions shall be taken to avoid sulphur contamination from atmosphere, apparatus, and reagents or other sources. Care should be taken not to allow sodium hydroxide reagent or apparatus 'wet' with this reagent to be exposed to laboratory atmosphere.

#### B-2 APPARATUS

**B-2.1 Reduction Apparatus**—The reduction apparatus shall be of the shape, dimensions and assembly as shown in Fig. 3. The cone and delivery tube is connected to a supply of nitrogen via a Dreschel bottle and the luted venting device. Rubber tubing shall not be used for nitrogen supply connection; PVC or

other sulphur-free plastics tubing is suitable. The 100-rol flash is heated electrically by means of a mantle. The apparatus consists of the following:

**B-2.1.1 Reduction Flask** — 100-ml round-bottomed flask with two short upright necks having respectively 10/19 and 14/23 ground-glass sockets (see IS 5165). The flask is also fitted with a 10/19 ground-glass socket inclined to centre-bottom.

**B-2.1.2 Glass Delivery Tube** — A 10/19 (see IS 5165) cone and stem with the end drawn out to a 1-mm hole, of such a length that the tip is within 5 mm of the centre of the bottom of the flask when the cone is in position in the inclined socket.

**B-2.1.3 Tap Funnel**, capacity 20 ml with 10/19 (see IS 5165) cone and socket.

**B-2.1.4 Adaptor**, right-angle connection with 10/19 cone.

**B-2.1.5 Condenser**— A Liebig condenser, effective length 150 mm, with a 14/23 cone and socket.

**B-2.1.6 Absorber** — A delivery tube of  $6 = 0.5$  mm outer diameter bent at  $110^\circ$  and containing a small expansion chamber in the upright section. The lower end of the tube has a 1 mm hole, and fits into a covered 200 mm  $\times$  32 mm outer diameter boiling tube. The upper end of the delivery tube is bent at an angle of  $70^\circ$  and fitted with a 14/23 cone at a distance of approximately 135 mm from the longer arm.

**B-2.1.7 Gas Washing Bottle**, Dreschel bottle having a dip-tube of about 6 mm outer diameter.

**B-2.1.8 Microburette**, capacity 10 ml arranged so that the liquid in the absorber can be titrated.

**B-2.2 Flask** — A 500 ml stoppered conical flask, marked at the 400 ml level with the cone and stopper lubricated with silicone grease.

**B-2.3 Measuring Cylinder**, capacity 10 ml.

**B-2.4 Pipette (Not to be Operated by Mouth)**

**B-2.5 Thermometer**, any suitable thermometer including the interval 75 to 80°C.

### B-3 REAGENTS

**B-3.1 Acetone**

**B-3.2 Propan-2-ol**

**B-3.3 Nitrogen**

**B-3.4 Raney Nickel**, 50 percent nickel, 50 percent aluminium.

**B-3.5 Sodium Hydroxide Solutions, 2.5 N and 1 N** — Clean the conical flask with nitric acid/potassium dichromate mixture [prepared by dissolving 5 g of potassium dichromate in 5 ml of water and adding 100 ml of concentrated (15 N) nitric acid, stirring continuously] ; rinse thoroughly with water. Fill with water to the 400 ml mark and add the mass of sodium hydroxide pellets appropriate for preparation of the 2.5 N or 1 N solution. Swirl gently until dissolution is complete and allow to cool.

**B-3.6 Hydrochloric Acid, 5N.**

**B-3.7 Potassium Hydroxide**, 40 g/l solution in ethandiol.

**B-3.8 Mercuric Acetate Solution** — Dissolve 0.675 g of mercuric oxide, previously dried at 100°C, in 50 ml of water containing 2 ml of glacial (17 M) acetic acid. Dilute to 1 000 ml with water and mix well. Dilute 50 ml of the solution thus prepared to 250 ml with water and mix well. One millilitre of the diluted solution is equivalent to 0.02 mg of sulphur.

**B-3.9 Dithizone Indicator Solution**, 1 g/l in acetone, prepared fresh daily, or every 3 days, if stored in a refrigerator.

NOTE — When experience has been gained with the concentration of indicator required, a few grains of the solid indicator may be added to the absorber. In this way any instability of indicator solution is overcome.

### B-4 PROCEDURE

**B-4.1** Clean the apparatus thoroughly with a mixture of nitric acid and potassium dichromate. Rinse thoroughly with water and acetone and dry in an oven which has not been contaminated with sulphur or sulphur containing materials in previous use. The apparatus is self scouring and, when in constant use, shall not be cleaned between determinations, except for rinsing the flask, delivery tube, absorber and thermometer with water. When not in constant use, it shall be cleaned between determinations with water and acetone.

NOTE — For activating nickel perfectly, the weighed Raney nickel is kept in caustic solution for at least 4 h.

**B-4.2** Weigh accurately about 0.5 g of Raney nickel and put it in the reduction flask using a cone made from glazed paper, and add 10 ml of sodium hydroxide solution (2.5 N) from the measuring cylinder.

Care shall be taken at this stage because there is a vigorous reaction.

When the reaction has subsided, swirl the liquid in the flask to bring the nickel adhering to the sides of the flask to the bottom. Set the flask aside for 10 min and then decant the supernatant liquid. Wash down both necks of the flask with 10 to 15 ml of water. Swirl the water vigorously to disturb the nickel residue, but avoid entrainment of air and, with minimum delay for settling, decant the water as completely as possible without too much attention to removing the last drop. Repeat the water wash three more times, and follow with a wash with 10 ml of propan-2-ol. Decant most of the propan-2-ol leaving enough to cover the catalyst, and add a further 10 ml of propan-2-ol.

NOTE — Incrustations around the stoppers and necks of sodium hydroxide bottles contain sufficient quantities of sulphur to affect test results. Such incrustations should be removed without allowing material to fall into the bottle. Before using solution from the bottle, pour a little to waste. Replace the stopper promptly.

**B-4.3** Assemble the apparatus except for the tap funnel, lightly greasing all the joints with silicone grease. Add 50 ml of a mixture of equal parts of sodium hydroxide solution (1 N) and acetone to the boiling tube and add 5 drops of the dithizone indicator solution.

**B-4.3.1** Calculate the appropriate size of sample as follows:

|   |
|---|
| 100 ml  |
| Specified or expected sulphur content ( mg/kg ) |

(with a maximum of 50 ml) and pipette this volume into the flask through the 10/19 socket.

**B-4.4** Complete the assembly of the apparatus. Measure 10 ml of the hydrochloric acid solution into the tap funnel. Pass nitrogen at the rate of 2 or 3 bubbles/second as shown in the Dreschel bottle containing the ethane-di-ol potassium hydroxide solution. Note the burette reading and titrate the contents of the absorber with the mercuric acetate solution to a pale pink colour.

**B-4.4.1** Heat the flask at such a rate that the contents boil gently in about 10 min. Maintain the heating for a further period of 30 min at such a rate that small bubbles rise copiously from the nickel and gentle refluxing occurs.

**B-4.4.2** Increase the input to the heating mantle slightly and allow the hydrochloric acid solution to drip slowly (10 ml in 5 to 10 min) into the flask. Vigorous generation of hydrogen will occur, but little or no hydrogen sulphide will be evolved until about half the hydrochloric acid has entered the flask. Titrate the absorbing solution to a pink colour. As hydrogen sulphide is evolved and absorbed and the colour of the absorbing solution reverts to yellow, titrate in more mercuric acetate solution in order to restore the pink colour. After the addition of the acid, open the tap of the funnel occasionally to sweep forward any hydrogen sulphide that may have collected below it. When the evolution of hydrogen sulphide has almost ceased, increase the nitrogen flow rate to about 5 bubbles/second in the bottle to improve the transfer of hydrogen sulphide to the absorber.

**B-4.4.3** When the evolution of hydrogen sulphide has apparently ceased, turn off the nitrogen temporarily and cool the flask by reducing the heat input and by blowing a little air on to it or by applying a damp cloth. The reduction in pressure will cause the absorbing solution to rise up the delivery tube. Restore the nitrogen flow before the absorbing solution reaches the bend above the cone. Repeat this operation at about 2 min intervals until no more hydrogen sulphide is washed down. If any liquid enters the cone, the test shall be abandoned.

**B-4.4.4** Boil the contents of the flask vigorously and continue the titration to the end point.

**B-4.4.5** Carry out a blank test on the reagents omitting the sample. Once a day is normally sufficient but the blank shall always be re-determined, if there is any change in the reagents, apparatus, or laboratory atmosphere which could conceivably affect the blank value. This value should not exceed 0.6 ml.

## B-5 CALCULATION

$$\text{Total sulphur content, mg/kg} = \frac{20(V_1 - V_2)}{V D}$$

where

$V_1$  = volume of mercuric acetate solution used for the sample titration, in ml;

$V_2$  = volume of mercuric acetate solution used for the blank test, in ml;

$V$  = volume of sample taken for the test, in ml; and

$D$  = density of the sample at the temperature at which it was measured, in g/ml.

## ANNEX C

[Table 1, Sl No. (vi)]

### DETERMINATION OF DISTILLATION RANGE

#### C-1 OUTLINE OF THE METHOD

The method includes distillation of 100 ml test portion under prescribed conditions, which are equivalent to simple batch distillation, systematic observation of thermometer readings and volumes of condensate and calculation of the results from these data with correction to standard atmospheric pressure.

#### C-2 DEFINITIONS

For the purpose of this Annex the following definitions shall apply:

**C-2.1 Initial Boiling Point** — The temperature noted (corrected, if required) at the moment when the first drop of condensate falls from the tip of the condenser

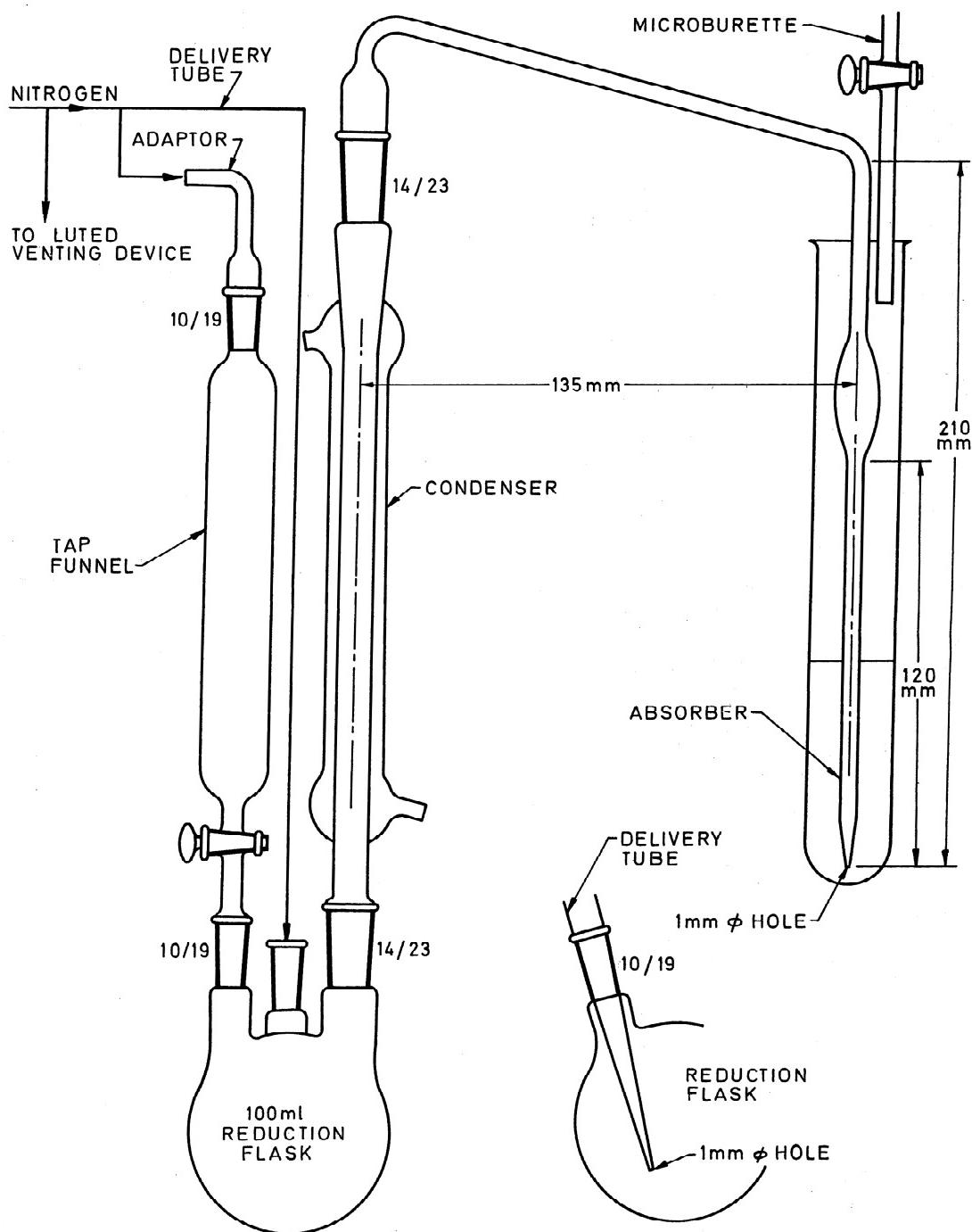


FIG. 3 TOTAL SULPHUR REDUCTION APPARATUS

during a distillation carried out under standardization conditions.

**C-2.2 Dry Point** — The temperature note (corrected, if required) at the moment of vaporization of the last drop of liquid at the bottom of the flask during a distillation carried out under standardized conditions, discarding any liquid on the side of the flask and on the thermometer.

**C-2.3 Boiling Range** — The temperature interval between the initial boiling point and dry point.

### C-3 APPARATUS

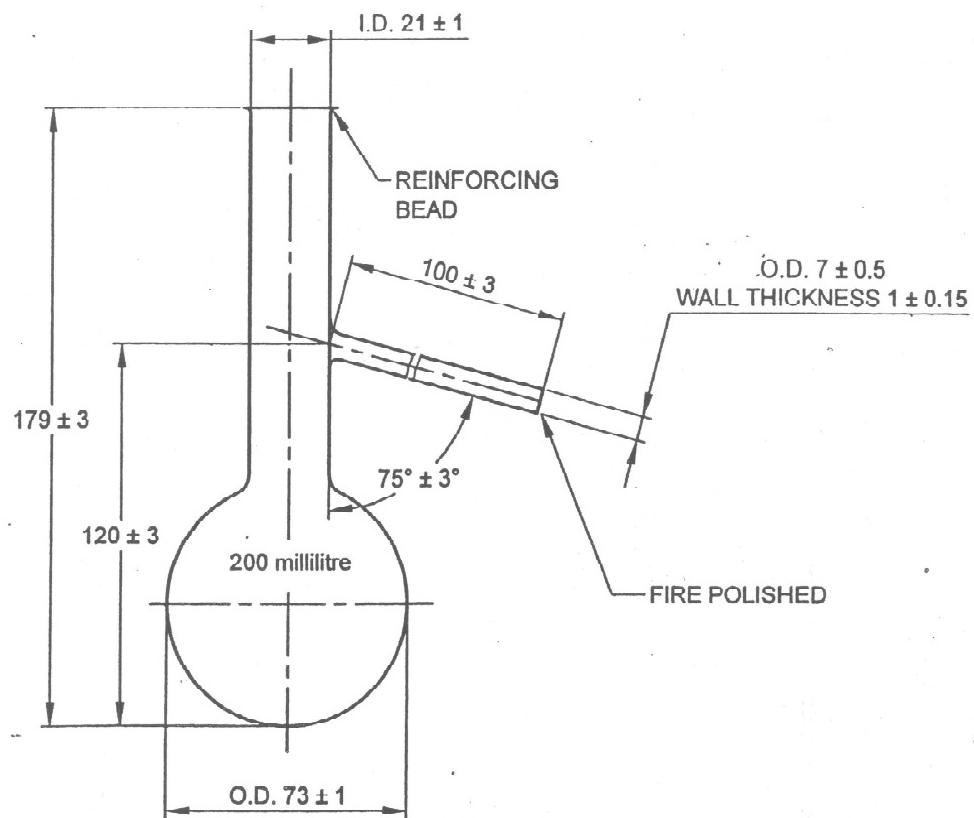
The apparatus, a suitable form of which is shown in Fig. 4 to 7, shall comprise the following items.

**C-3.1 Distillation Flask**, of heat resistant glass, of capacity 200 ml, conforming to the dimensions shown in Fig. 4.

**C-3.2 Thermometer**, mercury-in-glass type, nitrogen-filled, graduated on the stem, enamel-backed, and conforming to Table 5.

**Table 5 Requirements of Thermometer**

| SI No. | Characteristic  | Requirement        |
|--------|---|--------------------|
| (1)    | (2)   | (3)                |
| i)     | Immersion, mm   | 100                |
| ii)    | Range, °C   | 72-126             |
| iii)   | Graduation, °C  | 0.2                |
| iv)    | Longer lines at each, °C                                  | 1                  |
| v)     | Figured at each °C  | 2                  |
| vi)    | Scale error not to exceed, °C                             | 0.2                |
| vii)   | Overall length, mm  | $395 \pm 5$        |
| viii)  | Stem diameter, mm   | 6.0-7.0            |
| ix)    | Bulb length, mm   | 15-20              |
| x)     | Distance from bottom of bulb, mm<br>to 72 °C<br>to 127 °C | 125-145<br>335-360 |
| xi)    | Expansion chamber to allow heating to, °C                 | 150                |



All dimensions in millimetres.

FIG. 4 DISTILLATION FLASK

### C-3.3 Draught Screen

#### C-3.3.1 For Use with a Gas Burner

**C-3.3.1.1** The draught screen shall be rectangular in cross-section and open at the top and bottom. It shall have the dimensions shown in Fig. 5 and be made of sheet of metal of thickness approximately 0.8 mm.

**C-3.3.1.2** In each of the two narrower sides of the draught screen, there shall be two circular holes of diameter 12.5 mm, the centres of which are situated 25 mm above the base of the draught screen. These holes shall occupy the positions shown in Fig. 5.

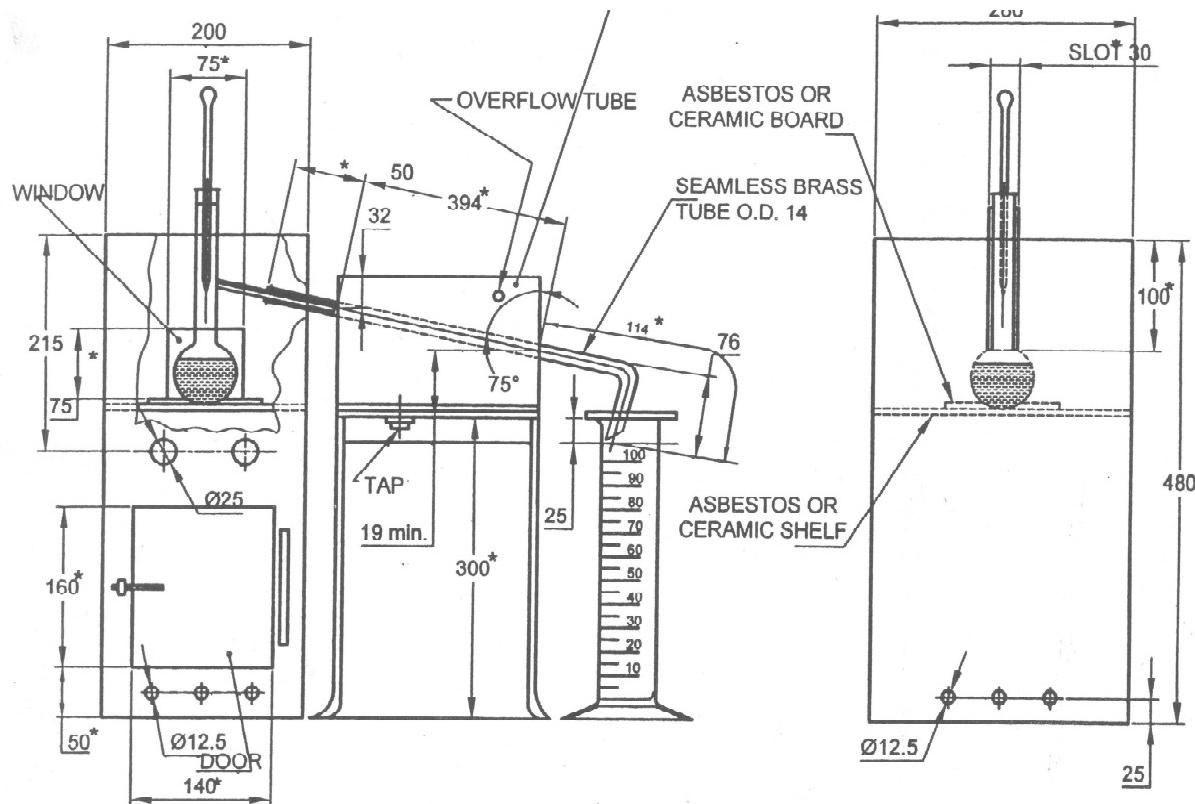
**C-3.3.1.3** At the middle of each of the wider sides, a vertical slot for the condenser tube, dimensioned approximately as shown in Fig. 5, shall be cut downwards from the top of the screen. A removable shutter of suitable dimensions shall be provided for closing whichever vertical slot is not in use. This

arrangement enables the condenser (see C-3.6) to be placed on either side of the draught screen.

**C-3.3.1.4** A shelf of ceramic material, of thickness 3 mm to 6 mm and possessing a centrally cut circular hole of diameter 75 mm to 100 mm, shall be supported horizontally in the screen and shall fit closely to the sides of the screen, to ensure that hot gases from the source of heat (see C-3.5) do not come in contact with the sides or neck of the flask (see C-3.1). The supports for this shelf may conveniently consist of triangular pieces of metal sheet firmly fixed to the screen at its four corners.

**C-3.3.1.5** A board as described in C-3.4 shall rest on this shelf.

**C-3.3.1.6** In one of the narrower sides of the screen, a door shall be provided having the approximate dimensions shown in Fig. 5 and overlapping the



\* Approximate dimensions given for guidance.

All dimensions in millimetres.

FIG. 5 DISTILLATION APPARATUS USING GAS BURNER

opening in the screen by approximately 5 mm all round.

### C-3.3.2 For Use with an Electric Heater

When an electric heater is employed, the portion of the draught screen above the shelf shall be as described in C-3.3.1, but the lower portion (including the shelf) may be modified or omitted, provided that the changes does not expose the distillation flask to draughts (*see Fig. 6*). Provision shall be made for adjustment of the shelf to facilitate fitting of the flask.

**C-3.4 Ceramic Boards**, of thickness 3 to 6 mm, with central holes of diameter 32 mm or 38 mm respectively and overall dimensions not less than 150 mm<sup>2</sup>. When a gas heater is employed, this board shall rest on the shelf described in C-3.3.1 when an electric heater is employed, the same arrangement shall be adopted, if the shelf is present; alternatively, the board may be placed directly on the heater or it may form the top of the heater. Provision shall be made for adjusting the height of the heater.

Whichever type of heater is employed, direct heat shall only be applied to the flask through the central hole in the ceramic board.

**C-3.5 Source of Heat**, comprising either a gas burner so constructed that sufficient heat can be obtained to distill the product at the uniform rate specified in C-5.3.2. A sensitive regulating valve or governor is desirable adjuncts or an electric heater capable of complying with the same requirements. (A heater of low heat retention, adjustable from 0 to 1 kW, has been found satisfactory.)

**C-3.6 Condenser**, of seamless brass tubing, of length 560 mm, outside diameter 14 mm and wall thickness 0.8 to 0.9 mm, surrounded by a metal cooling bath, preferably of copper or brass. The tube shall be set so that a length of approximately 390 mm is in contact with the cooling medium in the cooling bath, with about 50 mm outside the cooling bath at the upper end, and about 115 mm outside at the lower end. The length of the tube projecting at the upper end shall be straight and set at an angle of 75 to the vertical. The section of the tube inside the cooling bath may be either straight shall be 0.26 mm per linear millimetre of the condenser tube ( $\sin 15^\circ$ ), and no part of it shall have a gradient less than 0.24 mm nor more than 0.28 mm per linear millimetre of the tube. The projecting lower portion of the condenser tube shall be curved downward for a length of 76 mm and slightly backward so as to ensure contact with the wall of the receiver (*see C-3.7*) at a point 25 mm to 32 mm below the top of the receiver when it is in a position to receive the distillate. The lower end of the condenser tube shall be cut off at an acute angle

so that the tip may be brought into contact with the wall of the receiver.

The capacity of the cooling bath shall be not less than 5.5 litre of cooling medium. The arrangement of the tube in the cooling bath shall be such that its centre line is not less than 32 mm below the plane of the top of the bath at its point of entrance, and not less than 19 mm above the floor of the bath at its exist. Clearances between the condenser tube and the walls of the bath shall be at least 13 mm, except for the section adjacent to the points of entrance and exit.

The cooling bath may be provided with a tap at the bottom for drainage or inlet, and with an overflow tube near the top.

The main dimensions of the tube and cooling bath are shown in Fig. 5.

**C-3.7 Receiver**, of capacity 100 ml, complying with the details shown in Fig. 7. None of the graduation lines shall be in error by more than 1 ml. The shape of the base is optional but it shall be such that the receiver does not topple when placed empty on a surface inclined at an angle of 15 to the horizontal.

**C-3.8 Barometer**, accurate to the nearest 1 m bar, 0.1 kPa or 1 mmHg.

## C-4 ASSEMBLY AND PREPARATION OF APPARATUS

### C-4.1 Assembly

Assemble the apparatus, swabbing out the condenser with a piece of lint-free cloth attached to a wire cord or by any other suitable means, and paying attention to the following details:

- a) *Position and choice of thermometer* — Use the thermometer as indicated at C-3.2. Centre the thermometer into the neck of the flask through a tight-fitting silicone-rubber or cork stopper so that the upper end of the contraction chamber is level with the lower wide of the vapour tube at its junction with the neck of the flask.
- b) *Support for flask* — If a draught screen with ceramic shelf is used, place the appropriate ceramic board (*see C-3.4 and C-5.3*) on top of the shelf so that the two holes are concentric.
- c) *Connection of flask to condenser* — Make a leak-proof connection of the flask (*see C-3.1*) to the tube of the condenser (*see C-3.6*) by means of a tight-fitting silicone-rubber or cork stopper through which the vapour tube of the flask passes. Connect the flask to the

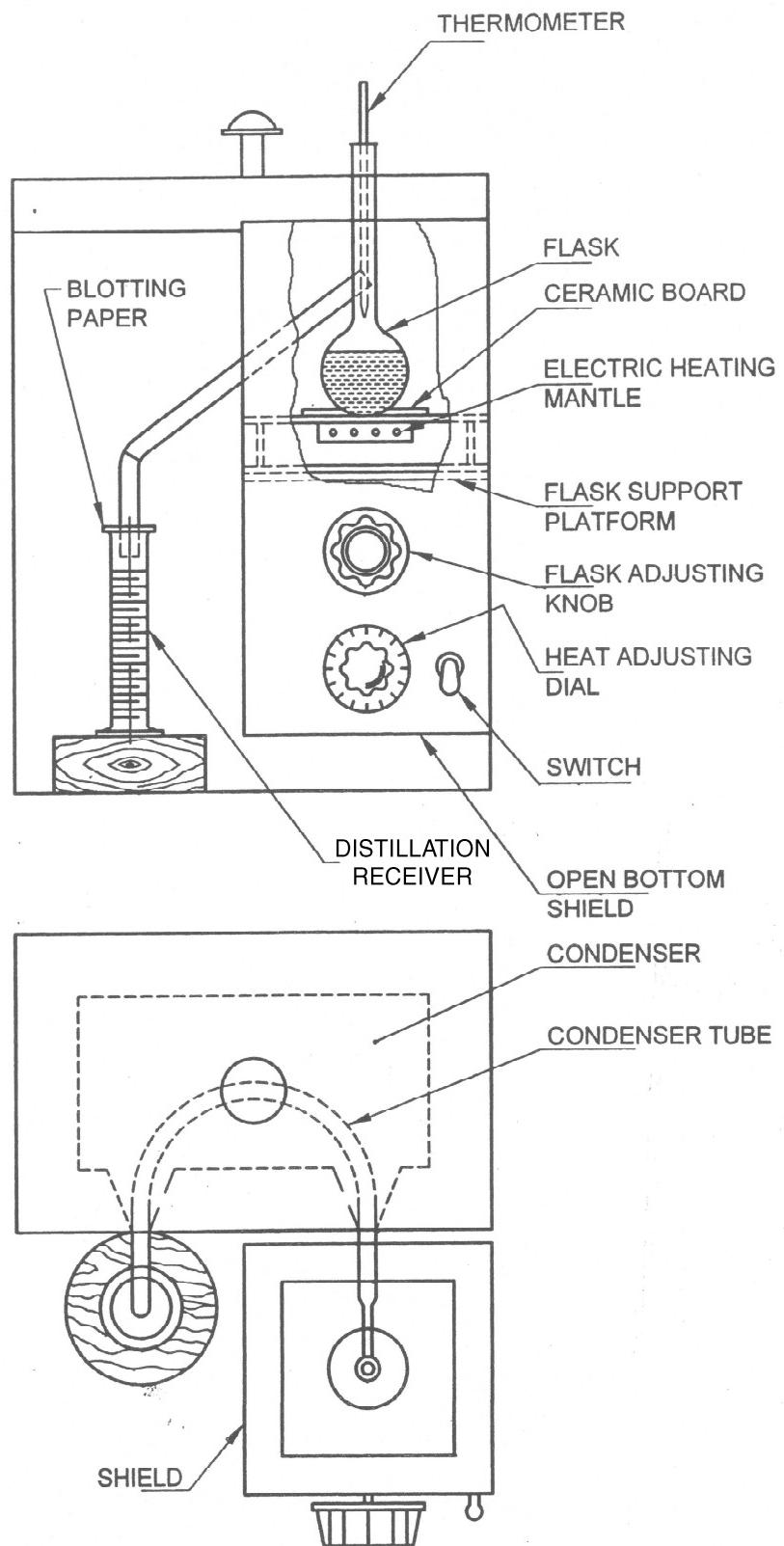


FIG. 6 DISTILLATION APPARATUS USING ELECTRIC HEATER



All dimensions in millimetre.

FIG. 7 DISTILLATION RECEIVER

condenser so that the flask is in a vertical position; the end of the vapour tube shall extend at least 25 mm and not more than 50 mm beyond the cork into the condenser tube and shall be co-axial with it.

Place the flask in such a position on the board that the base completely closes the hole in the board.

#### C-4.2 Filling of Cooling Bath

Fill the bath with water or with water and crack ice in sufficient quantity to cover the condenser tube, so as to ensure that the temperature of the bath at the start of and during distillation remains between 25°C to 30°C.

#### C-4.3 Adjustment of Temperature of Sample

Adjust the temperature of the sample to between 20°C

to 30°C, to prevent excessive evaporation of the product.

### C-5 PROCEDURE

#### C-5.1 Test Portion

Using the graduated receiver (*see C-3.7*), measure  $100 \pm 0.5$  ml of the sample at the temperature to which it has been adjusted as specified in **C-4.3**. Remove the flask (*see C-3.1*) from the apparatus and transfer the test portion directly to the flask, allowing the receiver to drain for 15 to 20 s. Do not allow any of the test portion to enter the vapour tube.

#### C-5.2 Apparatus Assembly

Connect the flask to the condenser (*see C-3.6*) and insert the thermometer (*see C-3.3*) as described

in C-4.1.1 and position the flask as described in C-4.1.3. Place the receiver (*see C-3.7*), without drying, at the outlet of the condenser tube in such a position that the condenser tube extends into the receiver at least 25 mm but does not extend below the 100 ml mark.

Place a flat cover on the top of the receiver to prevent entry of condensed moisture.

### C-5.3 Operating Conditions

A certain amount of judgement is necessary in choosing the best operating conditions to obtain acceptable accuracy and reproducibility. As a general guide it is recommended that the following conditions shall be established:

- a) *Flask support* — Hole diameter, 32 mm
- b) *Heating rate* — Time from application of heat to collection of first drop of distillate 5 to 10 min, and time for rise of vapour column in neck to flask to side arm, 2.5 min to 3.5 min.

### C-5.4 Initial Boiling Point

Record the temperature at the instant the first drop of distillate falls from the tip of the condenser as the initial boiling point (*see C-2.1*).

### C-5.5 Distillation

Adjust the heat input so that the distillation proceeds at a rate of 4 to 5 ml/min (approximately 2 drops per second), and move the receiver so that the tip of the condenser tube touches one side of the cylinder after the first drop falls.

### C-5.6 Dry Point

Without changing the heater setting, continue distillation beyond the 95 percent point until the dry point (*see C-2.2*) is observed. Record the temperature at this moment as the dry point. If a dry point is not obtained (that is, if active decomposition occurs before the dry point is reached, as shown by a rapid evolution of vapour or heavy fumes, or if there is liquid remaining on the bottom of the flask when the maximum temperature is observed in the distillation thermometer), record this fact.

### C-5.7 Atmospheric Pressure

Read and record the barometric pressure to the nearest 1 m bar, 0.1 kPa or 1 mmHg.

## C-6 CALCULATIONS

### C-6.1 Thermometer Bore Correction

Apply the correction for any variation in the bore of the thermometer as given by the calibration certificate.

### C-6.2 Thermometer Bulb Shrinkage Correction

Apply the correction for shrinkage of the mercury bulb of the thermometer as determined by any change in its ice or steam point, where applicable. Other means can be employed, such as the use of a platinum-resistance thermometer or a recognized standard thermometer.

### C-6.3 Barometer Correction

After applying the corrections for thermometer error, correct each reading for deviation of the barometric pressure from normal by adding algebraically the correction, calculated as follows:

$$K(760-p_0)$$

$$\text{or } K'(1\ 013-p_1)$$

$$\text{or } K'(1\ 013-p_2)$$

where

$K$  = rate of change of boiling point with pressure, in  $(^{\circ}\text{C}/\text{mmHg})$  (*see Table 6*);

$K'$  = rate of change of boiling point with pressure, in per mill bar or per 0.1 kPa  $(^{\circ}\text{C}/\text{mbar or } ^{\circ}\text{C}/\text{kPa})$  (*see Table 6*);

$p_0$  = barometric pressure, in mmHg, during the test;

$p_1$  = barometric pressure, in millibars, during the test; and

$p_2$  = the barometric pressure, in kilopascals, during the test.

**Table 6 Boiling Point**

| SI No.<br>(1) | Characteristic<br>(2)  | Requirement<br>(3) |
|---------------|--|--------------------|
| i)            | Boiling point $^{\circ}\text{C}$ (at 1 013 bar, 1 013 kPa, 760 mmHg)   | 110.6              |
| ii)           | Rate of change of boiling point with pressure $K'(^{\circ}\text{C}/\text{mbar}, ^{\circ}\text{C}/0.1 \text{ kPa})$ | 0.035              |
| iii)          | Rate of change of boiling point with pressure $K(^{\circ}\text{C}/\text{mmHg})$                                    | 0.046              |

## ANNEX D

[Table 1, Sl No. (vii)]

### DETERMINATION OF NON-AROMATIC HYDROCARBONS

#### D-1 OUTLINE OF THE METHOD

A known amount of an internal standard is added to the specimen that is then introduced into a gas chromatograph (GC) equipped with a flame ionization detector (FID). The peak area of each impurity and the internal standard is measured and the amount of each impurity is calculated from the ratio of the peak area of the internal standard *versus* the peak area of the impurity. Purity by GC is calculated by subtracting the sum of the impurities found from 100.00. Results are reported either in weight percent or volume percent.

#### D-2 APPARATUS

**D-2.1 Gas Chromatograph** — Any instrument having a flame ionization detector that can be operated at the conditions given in Table 7. The system should have sufficient sensitivity to obtain a minimum peak height response for 10 mg/kg *n*-butylbenzene of twice the height of the signal to background noise.

**D-2.2 Columns** — Both capillary and packed columns containing a stationary phase of cross-linked polyethylene glycol have been found satisfactory. The column must give satisfactory resolution of the internal standard from the solvent and the impurity peaks, and should be such that benzene is eluted between *n*-nonane and *n*-decane. Table 7 contains a description of a column that has been found satisfactory.

**D-2.3 Recorder**, electronic integration is recommended.

**D-2.4 Microsyringe**, 10, 50, and 500 µl capacity.

**D-2.5 Volumetric Flask**, 50 ml capacity.

#### D-3 REAGENTS

**D-3.1 Purity of Reagents** — Reagent grade chemicals shall be used in all tests, unless otherwise indicated.

**D-3.2 Carrier Gas**, chromatographic grade helium is recommended.

**D-3.3 High Purity *p*-Xylene**, 99.999 percent by weight or greater purity.

**D-3.3.1** Most *p*-xylene is available commercially at purity less than 99.9 percent and can be purified by recrystallization. To prepare 1.9 litre of high purity *p*-xylene, begin with approximately 3.8 litre of material and cool in an explosion-proof freezer at

$-10 \pm -5^{\circ}\text{C}$  until approximately  $\frac{1}{2}$  to  $\frac{3}{4}$  of the *p*-xylene has frozen. This requires about 5 h. Remove the sample and decant the liquid portion. The solid portion is the purified *p*-xylene. Allow the *p*-xylene to thaw and repeat the crystallization step on the remaining sample until the *p*-xylene is free of contamination as indicated by gas chromatography.

#### D-3.4 Pure Compounds

For calibration, shall include *n*-nonane, benzene, toluene, ethylbenzene, *o*-xylene and cumene. The purity of all reagents should be more than 99 weight percent. If the purity is less than 99 percent, the concentration and identification of impurities must be known so that the composition of the standard can be adjusted for the presence of the impurities.

##### D-3.4.1 Internal Standard

*n*-butylbenzene (NBB) is the recommended internal standard of choice, however, other compounds may be found acceptable provided they don't interfere.

**Table 7 Instrumental Parameters**  
(Clauses D-2.1, D-2.2 and D-4)

| Sl No.<br>(1) | Detector<br>(2)   | Flame Ionization<br>(3)                                    |
|---------------|---|--|
| i)            | Column:<br>a) Tubing<br>b) Stationary phase   | Fused silica<br>Crosslinked<br>polyethylene glycol<br>0.25 |
|               | c) Film thickness, µ<br>d) Length, m<br>e) Diameter, mm   | 60<br>0.32 ID  |
| ii)           | Temperatures:<br>a) Injector, °C<br>b) Detector, °C<br>c) Oven:<br>1) Initial °C<br>2) Time 1, min<br>3) Final, °C<br>4) Rate, °C/min<br>5) Time 2, min | 270<br>300<br>60<br>10<br>150<br>5<br>10                   |
| iii)          | Carrier gas<br>Flow rate, ml/min<br>Split ratio<br>Sample size, µl<br>Analysis time, min<br>Linear velocity @ $145^{\circ}\text{C}$ , cm/s              | Helium<br>1.0<br>100 : 1<br>1.0<br>30<br>20                |

#### D-4 PREPARATION OF APPARATUS

Follow manufacturer's instructions for mounting and conditioning the column into the chromatograph and adjusting the instrument to the conditions described in Table 7 allowing sufficient time for the equipment to reach equilibrium.

## D-5 CALIBRATION

**D-5.1** Prepare a synthetic mixture of high purity *p*-xylene with representative impurities. The volume of each hydrocarbon impurity shall be measured to the nearest 0.1  $\mu$ l and all liquid reference compounds shall be brought to the same temperature before mixing. Refer to Table 8 for an example of a calibration blend. The nonaromatic fraction is represented by *n*-nonane, while *o*-xylene represents the xylene fraction. Cumene represents the aromatic hydrocarbons containing nine carbon atoms or greater ( $C_9$  aromatics).

**Table 8 Preparation of Calibration Blend**  
(Clauses D-5.1 and D-5.2)

| SI No. | Compound                       | Density | Recommended Volume $\mu$ l | Resulting Concentration |                    |
|--------|--------------------------------|---------|----------------------------|-------------------------|--------------------|
|        |                                |         |                            | Volume Percent (5)      | Weight Percent (6) |
| (1)    | (2)                            | (3)     | (4)                        |                         |                    |
| i)     | <i>p</i> -xylene (see C-3.3.1) | 0.857   | 50.00                      | 99.72                   | 99.72              |
| ii)    | Benzene                        | 0.874   | 10.00                      | 0.020                   | 0.020              |
| iii)   | Toluene                        | 0.862   | 10.00                      | 0.020                   | 0.020              |
| iv)    | Ethylbenzene                   | 0.863   | 50.00                      | 0.100                   | 0.101              |
| v)     | <i>o</i> -xylene               | 0.876   | 50.00                      | 0.100                   | 0.099              |
| vi)    | Cumene                         | 0.857   | 10.00                      | 0.020                   | 0.020              |
| vii)   | <i>n</i> -nonane               | 0.714   | 10.00                      | 0.020                   | 0.017              |

**D-5.2** Using the exact volumes and densities in Table 8, calculate the weight percent concentration for each impurity in the calibration blend as follows:

$$C_i = [(D_i)(V_i)]/[(V_p)(D_p)](100)$$

where

- $D_i$  = density of impurity *i* from Table 8;
- $V_i$  = volume of impurity *i*, in ml;
- $D_p$  = density of *p*-xylene from Table 8;
- $V_p$  = volume of *p*-xylene, in ml; and
- $C_i$  = concentration of impurity *i*, weight percent.

**D-5.3** Into a 50 ml volumetric flask, add 50.0  $\mu$ l of *n*-butylbenzene (NBB) to 50.00 ml of the calibration blend and mix well. Assuming a density of 0.857 for the calibration blend and 0.856 for NBB, the resulting NBB concentration will be 0.100 weight percent, as determined from the equation given in **D-5.2**.

**D-5.3.1** All solutions and reference compounds must be brought to the same temperature, preferably 25°C, prior to adding the internal standard.

**D-5.4** Inject the resulting solution from **D-5.3** into the chromatograph and obtain a chromatogram.

**D-5.5** Determine the response factor for each impurity relative to NBB by measuring the area under each peak and calculate the response factor as follows:

$$RRF_i = (A_s)(C_i)/(C_s)(A_i)$$

where,

- $RRF_i$  = response factor for impurity *i* relative to the internal standard;
- $A_i$  = peak area of impurity, *i*;
- $A$  = peak area of the internal standard, NBB;
- $C_s$  = concentration of the internal standard, NBB, weight percent; and
- $C_i$  = concentration of impurity *i*, as calculated in **D-5.2**, weight percent.

## D-6 PROCEDURE

**D-6.1** Bring the internal standard and the sample to the analyzed to identical temperatures, preferably 25°C. Make sure that the temperature of the sample is consistent with that of the calibration standard prepared in **D-5**. Pipette 50  $\mu$ l of internal standard into a 50 ml volumetric flask containing 50 ml of sample. Mix well.

**D-6.2** Depending upon the actual chromatograph's operating conditions, inject an approximate amount of sample into the instrument. Make sure that the injection amount is consistent with those conditions used to meet the criteria in **D-6.1**.

**D-6.3** Measure the area of all peaks except the major component(s). Measurements on the sample must be consistent with those made on the calibration blend. The nonaromatic fraction includes all peaks up to toluene (except for the peak assigned as benzene). Sum together all the nonaromatic peaks and report as a total area. The  $C_9$  aromatic fraction includes cumene and all peaks emerging after *o*-xylene. Sum together all the  $C_9$  aromatic peaks and report as a total area.

## D-7 CALCULATIONS

**D-7.1** Calculate the weight percent concentration of the total nonaromatics and each impurity as follows:

$$C_i = (A_i)(RRF_i)(C_s)/(A_s)$$

**D-7.2** Calculate the volume concentration of the total nonaromatics, total  $C_9$  aromatics and each trace aromatic as follows:

$$V_i, \text{ percent} = (C_i)(D_i)$$

where

- $V_i$  = concentration of impurity *i*, volume percent, and
- $D_i$  = density of impurity *i* from Table 3.

**D-7.3** Calculate the total percentage of non-aromatic hydrocarbons by adding the percentage of individual components  $V_i$  as follows:

$$\text{Non-aromatic hydrocarbons, volume percent } V_i = \sum V_i$$

## ANNEX E

[Table 1, Sl No. (xi)]

### DETERMINATION OF THE PRESENCE OF HYDROGEN SULPHIDE AND SULPHURDIOXIDE

#### **E-1 OUTLINE OF THE METHOD**

This test method involves a qualitative colour test for H<sub>2</sub>S and SO<sub>2</sub> that utilizes filter paper containing lead acetate and starch paper containing potassium iodate. The test is performed when carrying out the Test for distillation of aromatic hydrocarbons as per Annex B.

#### **E-2 REAGENTS**

**E-2.1 Lead Acetate Solution**, saturated.

**E-2.2 Potassium Iodate Solution (100 g/l)**— Dissolve 10g of potassium iodate (KIO<sub>3</sub>) in water and dilute to 100 ml.

**E-2.3 Starch Paper** — Dip strips of filter paper in starch solution and dry.

#### **E-3 PROCEDURE**

Make a qualitative test for H<sub>2</sub>S and SO<sub>2</sub>, at the time of performing the distillation test (*see Annex C*). This is done by hanging a strip of filter paper moistened with the lead acetate solution and a strip of starch paper moistened with the potassium iodate solution on the end of the condenser tube. The strips are placed so that they are suspended in the upper part of the receiving cylinder so that drops of condensate pass between the strips without touching them. If, at the end of the test, the lead acetate paper shows discolouration, H<sub>2</sub>S is present but not SO<sub>2</sub>. If the lead acetate paper shows no discolouration but the starch iodate paper develops a blue colour, SO<sub>2</sub> is present but not H<sub>2</sub>S. If none of two papers show discolouration, neither H<sub>2</sub>S nor SO<sub>2</sub> is present.

## ANNEX F

*(Clause 6)*

### SAMPLING OF TOLUENE

#### **F-1 GENERAL REQUIREMENTS OF SAMPLING**

**F-1.1** In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed:

- a) Samples shall not be taken in an exposed place;
- b) Sampling instrument shall be clean and dry and shall be made of low or reduced spark generating material;
- c) Samples, the material being sampled, the sampling instrument and the containers for samples shall be protected from adventitious contamination;
- d) To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by shaking or stirring or both, or by rolling, so as to bring all portions into uniform distribution;
- e) Samples shall be placed in suitable, clean, dry and air-tight glass containers preferably of amber or blue colour;

- f) Sample container shall be sealed air-tight with a suitable stopper after filling and marked with full details of sampling, such as the date of sampling, the year of manufacture of material, the batch number, the name of the sample, etc. Particular care shall be taken to ensure that sealing methods do not contaminate the sample; and
- g) Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature.

#### **F-1.2 Additional Precautions**

The following additional precautions shall be observed:

- a) Rubber stoppers or composition corks shall not be used for closing the sample bottles;
- b) Sealing wax or other plastic material, if used, shall be applied in such a way that it does not contaminate the sample when the bottles are opened; and
- c) Each sample container shall be protected by covers of oil-proof paper, metal foil, viscose or other suitable impervious material over the

stopper to keep away moisture and dusts from the mouth of the bottle and to protect it while being handled.

## F-2 SAMPLING INSTRUMENT

**F-2.0** The following forms of sampling instrument may be used:

- Sampling bottle or can for taking samples from various depths in large tanks; and
- Sampling tube.

### F-2.1 Sampling Bottle or Can

It consists of a weighed bottle or metal container with removable stopper or top, to which is attached a light chain (*see Fig. 8*). The bottle or can is fastened to a suitable pole. For taking a sample, it is lowered in the tank to the required depth, and the stopper or top is removed by means of the chain for filling the container.

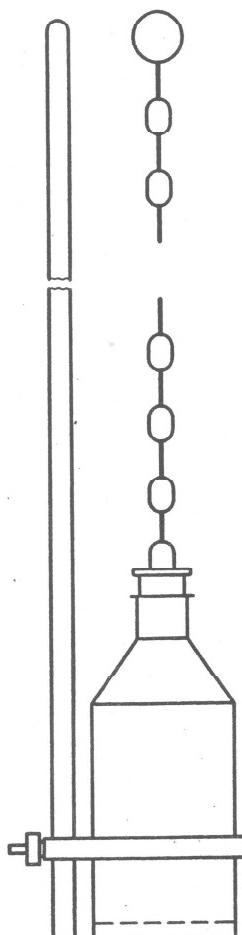


FIG. 8 SAMPLING BOTTLE OR CAN

### F-2.2 Sampling Tube

It is made of metal or thick glass and is about 20 to 40 mm in diameter and 400 to 800 mm in length (*see Fig. 9*). The upper and lower ends are conical and reach 5 to 10 mm internal diameter at the narrow ends. Handling is facilitated by two rings at the upper end. For taking a sample, the apparatus is first closed at the top with the thumb or a stopper and lowered until the desired depth is reached. It is then opened for a short time to admit the material and finally closed and withdrawn.

For small containers, the size of the sampling tube may be altered suitably.

## F-3 SCALE OF SAMPLING

### F-3.1 Lot

In a single consignment, all the containers of the same size and drawn from the same batch of manufacture shall constitute a lot. If a consignment is known to consist of containers of different sizes or of different batches of manufacture, then the containers belonging to the same size and batch of manufacture shall be grouped together and each such group shall constitute a separate lot. In case the consignment is in large tanks or vessels, the tanks or vessels belonging to the same batch of manufacture shall constitute a lot.

For ascertaining the conformity of the lot to the requirement of the specification, tests shall be carried out for each lot separately.

### F-3.2 Sampling from Containers

The number of containers to be selected for sampling shall depend on the size of the lot and shall be in accordance with Table 9.

**Table 9 Scale of Sampling**  
(Clause F-3.2)

| SI No.<br>(1) | Lot Size<br>(2) | No. of Containers to be Selected<br>(3) |
|---------------|-----------------|---|
| i)            | Up to 100       | 5                                       |
| ii)           | 101 to 200      | 6                                       |
| iii)          | 201 to 300      | 7                                       |
| iv)           | 301 to 400      | 8                                       |
| v)            | 401 to 500      | 9                                       |
| vi)           | 501 and above   | 10                                      |

NOTE — In the case of very small lots where the selection of the five containers may be uneconomical, all the containers shall be selected.

**F-3.2.1** The containers shall be selected at random. In order to ensure the randomness of selection, procedure given in IS 4905 may be adopted.

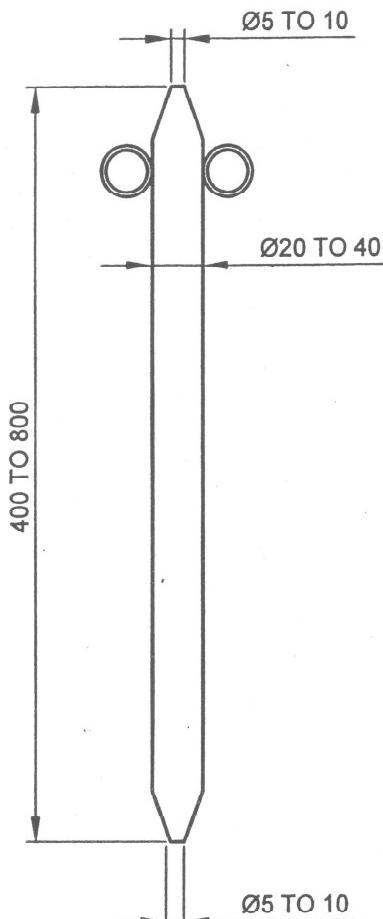
### F-3.3 Sampling from Tanks or Vessels

Each of the tanks or vessels in the lot shall be sampled separately for determining the conformity of the lot to the requirements of the standard.

## F-4 PREPARATION OF THE TEST SAMPLES

### F-4.1 Test Samples from Containers

To ensure that the sample taken from each container is fairly representative, the containers shall be mixed thoroughly, when possible, by shaking or stirring or rolling. Draw small samples of the material from various depths with the help of the sampling tube (*see Fig. 9*). The approximate quantity of the material to be drawn from a container shall be nearly equal to thrice the quantity required for testing purposes as indicated in F-5.1.



All dimensions in millimetres.

FIG. 9 SAMPLING TUBE

**F-4.1.1** Out of the material drawn from individual containers, a small but equal quantity of material shall be taken and thoroughly mixed to form a composite sample, sufficient for carrying out triplicate determinations for all the characteristics specified under F-5. The composite sample shall be divided into three equal parts, one for the purchaser another for the supplier and the third for the referee.

**F-4.1.2** The remaining portion of the material from each container shall be divided into 3 equal parts, each forming an individual sample. One set of individual samples representing the containers selected shall be for the purchaser, another for the supplier and the third for the referee.

**F-4.1.3** All the individual and composite samples shall be transferred to separate sample containers. These containers shall then be sealed airtight with stoppers and labeled with full identification particulars given in F-1.1 (f).

**F-4.1.4** The referee test sample, consisting of a composite sample and a set of individual samples, shall bear the seals of both the purchaser and the supplier. They shall be kept at a place agreed to between the purchaser and the supplier, to be used in case of any dispute.

### F-4.2 Test Samples from Tanks or Vessels

**F-4.2.1** For drawing a sample from a tank or vessel, lower the closed sampling bottle or can (*see F-2.1*) slowly to the required depth, open and fill it at that depth. Three samples shall be obtained at levels of one-tenth of the depth of the liquid from the top surface (top sample), one half of the depth (middle sample) and nine-tenths of the depth of the liquid from the top surface (lower sample). All the three samples thus obtained from a tank/vessel shall be mixed together in a clean dry container, and shall be divided into three parts, one for the purchaser, another for the supplier and the third for the referee. Each of the tanks or vessels in the lot shall be sampled in the above manner and separate samples obtained for each of the tanks or vessels. The approximate quantity of the material to be drawn from a tank or a vessel shall nearly be equal to thrice the quantity required for carrying out tests for all the requirements prescribed in F-5.

**F-4.2.2** All the samples thus obtained from the tanks or vessels in the lot shall be transferred to separate sample containers. These containers shall then be sealed air-tight with stoppers and labelled with full identification particulars given in F-1.1 (f).

**F-4.2.3** The referee test samples consisting of the

samples from the tanks or vessels in the lot, shall bear the seals of both the purchaser and the supplier. They shall be kept at a place agreed to between the purchaser and the supplier, to be used in case of any dispute.

## F-5 NUMBER OF TESTS

### F-5.1 For Samples from Containers

**F-5.1.1** Tests for the determination of distillation range and residue on evaporation shall be conducted on each of the individual samples separately (*see F-4.1.2*).

**F-5.1.2** Tests for the determination of all other characteristics given in Table 1 shall be conducted on the composite samples separately (*see F-4.1.1*).

### F-5.2 For Samples from Tanks or Vessels

Tests for the determination of all the characteristics given in Table 1 shall be conducted on the samples from different tanks or vessels separately.

## F-6 CRITERIA FOR CONFORMITY

### F-6.1 For Containers

#### F-6.1.1 For Individual Samples

The lot shall be declared as conforming to the requirements of the distillation range if test results for each of the individual samples tested in respect of distillation range satisfy the requirements as given in Table 1.

#### F-6.1.2 For Composite Samples

In respect of all other characteristics, the lot shall be considered as conforming to the composite sample satisfies each one of these requirements.

### F-6.2 For Tanks or Vessels

The lot shall be declared as confirming to the standard requirements of various characteristics, if each of the test results satisfies the relevant requirements specified in the standard individually.

*(Continued from second cover)*

ASTM D 4052 : 1996 Density and relative density of liquids by digital density meter

ASTM D 3120, ASTM D 4045, ASTM D 5453 test methods are available for the characteristic, total sulphur, stated and in case of dispute the corresponding Annex B, as given in Table 1 shall be the referee test method.

The following test methods are available for the characteristics stated in Table 1.

| <i>Characteristic</i>                           | <i>Alternate Method of Test</i> |
|---|---------------------------------|
| Toluene, benzene and C <sub>8</sub> + Aromatics | ASTM D 2360                     |
| Thiophene content                               | ASTM D 4375                     |

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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This Indian Standard has been developed from Doc No.: PCD 3 (2310).

### **Amendments Issued Since Publication**

| Amend No. | Date of Issue | Text Affected |
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